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ANALYTICAL AND EXPERIMENTAL EVALUATION OF JOINING SILICON NITRIDE
TO METAL AND SILICON CARBIDE TO METAL FOR ADVANCED
HEAT ENGINE APPLICATIONS

S. Kang, J. H. Selverian, D. O'Neil, H. Kim, and K. Kim

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ABSTRACT

This report summarizes the results of Phase II of Analytical and Experimental Evaluation of Joining Silicon Nitride to Metal and Silicon Carbide to Metal for Advanced Heat Engine Applications. A general methodology was developed to optimize the joint geometry and material systems for 650°C applications. Failure criteria were derived to predict the fracture of the braze and ceramic. Extensive finite element analyses (FEA) were performed to examine various joint geometries and to evaluate the affect of different interlayers on the residual stress state. Also, material systems composed of coating materials, interlayers, and braze alloys were developed for the program based on the chemical stability and strength of the joints during processing and service. The FEA results were compared with experiments using two methods; 1) an idealized strength relationship of the ceramic, and 2) a probabilistic analysis of the ceramic strength (NASA CARES). The results showed that the measured strength of the joint reached 30-80% of the strength predicted by FEA. Also, potential high-temperature braze alloys were developed and evaluated for the high-temperature application of ceramic-metal joints.

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1. INTRODUCTION

Phase I of this work [1] dealt with identifying the material systems and demonstrating analytical tools for use in designing ceramic-metal joints. This required the expression of the stress response of joints as a function of the mechanical and physical properties of the ceramic, metal, and other materials used in the joint. Various constitutive equations were derived for the joint materials as a function of temperature. Several joint geometries and interlayer materials were screened using the constitutive equations with the FEA technique.

The application temperatures of interest for Si_3N_4 to metal and SiC to metal joints were 650°C and 950°C . Significant efforts were made to develop material systems which could perform at these temperatures. The efforts in material systems development were divided into three parts: (1) coating materials development, (2) interlayer development, and (3) brazing alloy development. Based on the screening studies of coating materials, interlayer materials, and braze alloys, promising materials were tested as systems in terms of high-temperature performance and chemical stability during processing and service. The final screening was done using the final joint geometry suggested by the FEA study.

Performance tests of the brazed joints were done with the final geometry and material systems using room-temperature and 650°C torsion tests. These testing results were compared to the FEA predictions of the residual stress using a deterministic failure theory. Attempts were made to link the experimental results with the FEA predictions based on several simplifying assumptions. Thermal and mechanical fatigue testing was also done with the final joint systems.

In Phase II the material systems and joint geometry from Phase I was used for FEA study. The joint geometry was a cup joint with an interlayer and a bond area of 2 cm^2 . Silicon nitride was brazed to Inconel 718 and Incoloy 909 superalloy shafts using either a molybdenum or nickel interlayer and a Au-5Pd-2Ni braze alloy.

Finite element analysis and statistical failure analysis techniques were used to evaluate the joint. The residual stress state at room-temperature was calculated in the joints after the brazing cycle and was used for the statistical failure analysis of the silicon nitride component of the joints. The effect of

interlayer materials, nickel and molybdenum, on the residual stress state of a cup joint with an interlayer was evaluated. The nickel interlayer resulted in lower residual stresses in the silicon nitride at room-temperature compared to the molybdenum interlayer. The relative effect of material properties on the residual stress state of the joint is also discussed.

Once the stress state was known a failure criterion was needed to determine if the joint would survive. The selection of a failure criterion was complicated by the lack of a widely accepted model of ceramic fracture. Ceramics exhibit strength variations due to a distribution of microstructural flaws, whose exact size, shape, and orientation are unknown. This makes it difficult to apply a deterministic approach, used for metal systems and used in Phase I, to the design of ceramic components. Therefore, the CARES computer code was used for a statistical treatment of the expected failure mode was used to predict the reliability of these ceramic-metal joints [2,3] and the failure theory of Batdorf and Crose [4] was used.

Batdorf and Crose developed a fracture model based on a combination of linear elastic fracture mechanics and weakest link theory. Their approach was to consider a ceramic as a homogeneous isotropic brittle material with randomly oriented preexisting cracks. Crack growth and interactions were neglected. Failure was assumed to be predominantly controlled by the stress acting normal to a crack, and failure occurred when a critical stress was reached. The effect of compressive stresses on failure was assumed to be negligible. The distribution of preexisting cracks was obtained by fitting mechanical testing data to a functional equation. Their approach was encoded in the statistical treatment used here [2,3].

The CARES code was used in conjunction with finite element analysis to predict a probability (Weibull) plot of the brazed joint strength. This probability plot predicted the joint behavior at intermediate strength levels but was less accurate at the upper and lower ends of the probability distribution.

A new failure theory applicable to bimaterial systems was developed and applied to the joints in this study. The new failure theory accounts for the plasticity of the braze in reducing the stress in the ceramic near the ceramic/braze interface. The use of this theory results in the stresses in the ceramic being slightly reduced and results in an higher predicted strength of the joint.

The practical problems encountered in Phase I in joining two dissimilar materials were not only the thermal mismatch, which causes a significant residual stress at the interface, but compatibility among the joint components and its performance at high temperature. The joints made for Phase I of the program resulted in excellent performance at room-temperature in terms of torsional strength and mechanical fatigue. Thermal fatigue performance at 650°C was satisfactory. However, the joints in Phase I did not provide any torsional strength at 650°C. This renewed the need of new braze alloys and interlayer materials that can meet the high-temperature requirements for ceramic-metal joints.

Therefore, for the development of joining material systems, extensive efforts were made to design new high-temperature brazing alloys in Phase II. The design emphasis for the braze alloys was on (1) wettability, (2) melting temperature, (3) room-temperature and high-temperature mechanical properties, and (4) compatibility with other joining components. Master alloys were selected based on the requirements of the joints during brazing and applications under the guidance of phase diagram study. It is to be noted that the application temperature of interest for Si_3N_4 to metal joints was adjusted to 650°C.

Candidate compositions were evaluated with simple screening tests such as wetting test and hardness test. Simple bonding tests were also performed with coupon samples using those braze alloys which showed desirable properties. The most successful compositions were further modified and refined by incremental changes in the ratio between individual elements. Final compositions were cast into a bar and rolled into foils. Optimum brazing conditions were developed for different joint geometries by measuring joint strengths as a function of brazing temperature and time at temperature. Cylindrical joints employed in Phase I were used to measure high-temperature creep rate, room-temperature torsional joint strength, and thermal and mechanical fatigue properties. Finally the joint performance was interpreted with relevant microstructures of ceramic/braze interface for further improvement in alloy design.

2. DESIGN AND ANALYSIS OF METAL-CERAMIC JOINTS

2.1 Joint Materials

The ceramic was HIP'ed silicon nitride ($\text{Si}_3\text{N}_4 + 6\%\text{Y}_2\text{O}_3$). Two commonly used high-temperature metal structural alloys were used, Incoloy 909 (Fe-38.2Ni-13.0Co-4.7Nb-1.5Ti-0.03Al, in wt%) and Inconel 718 (Ni-20Cr-17Fe-5Nb-3Mo-1Ti-0.8Al, in wt%) both from Inco Alloys International. Incoloy 909 and Inconel 718 will be referred to as the structural alloy. Commercially pure nickel (nickel 200, 99.6 wt% Ni) was used as the nickel interlayer material. A powder metallurgy grade Mo (MT-104, Mo + 0.55-0.40 wt% Ti, GTE Products Corporation) was used as the molybdenum interlayer material. Nickel 200 and molybdenum MT-104 will be referred to as nickel (Ni) and molybdenum (Mo). Nickel was selected for its low flow stress, while Mo was selected for its low coefficient of thermal expansion. The braze alloy was Au-5Pd-2Ni, in wt%. The mechanical properties of a nickel aluminide were also used as a braze alloy in the finite element studies to investigate the effects of a high strength braze alloy [5]. The mechanical properties of these materials are listed in Appendix A.

2.2 Method of Comparing Different Joint Geometries

It was desired to have a single scalar "performance factor" for each geometry to be used as a method of comparison. The stress distribution in the joint was not used because there are synergistic effects of stress and volume on the fracture behavior of ceramics, making comparisons between different stress states difficult. The probability of survival (P_s) for the entire ceramic component was selected as the basis for comparing the different joint geometries. The P_s of the ceramic can represent the probability of joint failure since the metal components were not expected to fail, due to their high toughness. This parameter took into account the spatial distribution of the residual stresses, the strength distribution in the ceramic, and the effect of material volume on the likelihood of failure.

The evaluation of the probability of survival was done on an element basis. The probability of survival of each element was calculated and the individual values were multiplied together to obtain the $P_s v$ for the entire joint. $P_s v$ refers to the probability of survival based on volume flaws, the

subscript "V" is not included in the remainder of this report. A P_s value of 1 indicates all parts will survive, while a P_s of 0 indicates all parts will fail. The probability of survival of each element depended on the applied multiaxial stresses, element volume, shear-insensitive Batdorf crack density coefficient ($\bar{k}_B V$), and the normalized Weibull scale parameter ($\sigma_0 V$). The Batdorf crack density coefficient and the normalized Weibull scale parameter were calculated from the mechanical properties of the ceramic material.

2.3 Joint Geometries

Three joint geometries were studied. The butt joint, cup joint, and the cup joint with an interlayer are shown in Figure 2.1. A brazed joint area from 1.5 to 2 cm² was desired. The butt joint was chosen for its simplicity while the cup joint was selected because it can transmit higher torque as compared to the butt joint. The ceramic shaft had a radius of 0.7 cm for the joints without an interlayer and a brazed area of 1.5 cm². The ceramic shaft had a 0.63 cm radius for the cup joint with an interlayer and a brazed area of 2 cm², Figure 2.1c. The dimensions of the metal shaft and the braze were chosen so that the limit torque of the metal shaft was not less than what the braze layer could support. For the butt and cup joints the material system used was: silicon nitride, Au-5Pd-2Ni braze alloy, and Incoloy 909 structural alloy. For the cup joint with an interlayer several different materials were used for the structural alloy, braze alloy, and interlayer, and are described along with the results below.

Figure 2.1. Schematics of the (a) butt joint, (b) cup joint, and (c) cup joint with an interlayer. One-half of the cross-section of each axisymmetric joint is shown. The heavy arrows indicate the regions of lowest P_s .

2.4 Finite Element Analysis

The ABAQUS finite element code was used to calculate the residual stress state in the joints after the brazing cycle [6]. ABAQUS is especially well suited for solid mechanics problems involving large elasto-plastic deformations in three dimensions. The thermally expanded geometry of each joint at the brazing temperature (1180°C) was meshed using 6- and 8-noded quadratic, axisymmetric elements.

The brazed joint was cooled to 20°C, causing interference between the components. This interference resulted in permanent deformation of the braze layer, and residual stresses in the metal, braze, and ceramic. The stresses at room-temperature were reported at the nodes, integration points, and centroids of the elements. Also, volumes for each of the elements were computed for statistical failure analysis.

2.5 Probability of Survival of the Joints

The results from the finite element analyses of the different joint geometries were calculated for mechanical equilibrium at 20°C. The calculated stresses were input to the CARES program and used to calculate the probabilities of survival (P_S) of the various joints [2,3].

The failure analyses of the joints were based on element stresses and their volumes calculated by finite element analysis. The CARES program calculated the fast fracture reliability of ceramic components subject to complex loadings. CARES includes a number of fracture theories to predict the material response.

A failure analysis was performed on the joint geometries on the assumption that volume flaws controlled the ceramic failure. The Shetty criterion with penny-shaped volume cracks and K_I/K_{II} mixity parameter of 0.82 [4,7] was used in this analysis. A Weibull modulus of 15.6 and a characteristic strength of 970 MPa were used to calculate a shear-insensitive Batdorf crack density coefficient ($\bar{K}_{BV}$) of 32.2, and a normalized Weibull scale parameter (σ_{0V}) of $471.35 \text{ MPa(m)}^{3/15.6}$ [2]. Volume flaw analysis was performed on the three prospective ceramic-metal braze joint geometries.

2.5.1 Butt and Cup Joints

The regions of lowest P_s for the butt and cup ceramic-metal joint geometries are shown in Figure 2.2. These enlarged regions correspond to the regions highlighted by the heavy arrows in Figures 2.1a and 2.1b. These P_s results are valid only for the ceramic component in the joint, and were reported for the stresses occurring at 20°C after cooling from 1180°C. The cup joint was predicted to fail whereas only 130 in 10,000 butt joints are predicted to fail.

The probability of survival contour plots indicate the likely regions of crack initiation in the ceramic. There was a close relationship in the spatial distributions of the extreme values of the maximum principal stress distributions and the low P_s contours on the probability of survival plots. Therefore, the maximum principal stress, σ_1 , was considered to be a good indicator of possible failure locations in the ceramic.

The outside of the ceramic in the butt joint was sensitive to fracture because of the tensile stresses created by shrinkage of the metal alloy during cool-down from the brazing temperature (arrow in Figure 2.1a). The probable crack initiation site for the cup joint occurred at the end of the metal cylinder where it was joined to the ceramic (arrow in Figure 2.1b). A likely cause for this was the tendency for the metal component to pull on the ceramic at this point resulting in a large tensile stress

2.5.2 Cup Joints with Interlayers

The cup geometry was more desirable than the butt geometry because of the higher torque carrying capacity of the cup geometry. Therefore, to increase the survival rate of the cup joint an interlayer was placed between the ceramic and outer structural alloy to control the thermal expansion mismatch stresses. Nickel (low flow stress and high thermal expansion) and Mo (high flow stress and low thermal expansion) interlayers were studied to determine their effectiveness in reducing the residual stress transmitted to the ceramic. This joint is shown in Figure 2.1c.

The fracture analysis predicted survival of the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$, $\text{Si}_3\text{N}_4/\text{Mo}/\text{Incoloy 909}$, and $\text{Si}_3\text{N}_4/\text{Mo}/\text{Inconel 718}$ cup joints after the brazing cycle, Table 2.1. The lowest probability of survival was reported at the ends of the interlayer where the interlayer meets the ceramic (heavy arrow in Figure 2.1c). Again, the maximum principal stress was a good indicator of likely areas

of low probabilities of survival.

Figure 2.3 shows the probability of survival plot in the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ cup joint. Similar joints with different materials systems had very similar P_s contours, differing only in the magnitude of P_s . The Ni interlayer resulted in a higher P_s as compared to the Mo interlayer. The maximum tensile stress in the ceramic in the joint with a Ni interlayer was approximately one-half of the tensile stress in the ceramic in the joint with a Mo interlayer.

Table 2.1. Probability of failure for the ceramic component of ceramic-metal brazed joints. A P_s of 1.0 means all of the joints will survive, a P_s of 0.0 means all of the joints will fail.

Joint Geometry	Probability of Survival, P_s	Number of Failures per 10,000
Butt	0.987	130
Cylindrical lap joint (no interlayer)	0.000	10,000
Cylindrical lap joint with Incoloy 909 and Ni interlayer	0.999	10
Cylindrical lap joint with Incoloy 909 and Mo interlayer	0.997	30
Cylindrical lap joint with Inconel 718 and Mo interlayer	0.996	40

Figure 2.2. Probability of survival, P_s , in the (top) butt joint, the scale runs from 1.000 to 0.987. (bottom) cup joint, the scale runs from 1.000 to 0.000. A P_s of 1.0 means the parts survives, a P_s of 0.0 means the part fails. The metal is at the bottom and the ceramic is at the top.

Figure 2.3. Probability of survival, P_s , at 20°C in the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ joint superimposed on the deformed geometry. The P_s plots for the $\text{Si}_3\text{N}_4/\text{Mo}/\text{Incoloy 909}$ and $\text{Si}_3\text{N}_4/\text{Mo}/\text{Inconel 718}$ joints were similar in spatial distribution but the magnitude of P_s was larger, see Table 2.1.

2.6 Effect of Interlayer and Braze Properties on Residual Stress in Cup Joints with Interlayers

Figures 2.4 - 2.6 show the resultant maximum principle stress contours at 20°C plotted on the deformed mesh for the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$, $\text{Si}_3\text{N}_4/\text{Mo}/\text{Incoloy 909}$, and $\text{Si}_3\text{N}_4/\text{Mo}/\text{Inconel 718}$ brazed joints, respectively. Figure 2.7 shows contours of the total plastic strain at 20°C plotted on the deformed mesh for the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ brazed joint. Here, the total plastic strain is defined as the total accumulation of plastic strain occurring during cool-down from 1180°C to 20°C.

Figures 2.4 - 2.7 serve to illustrate several important points. There are two main components of the stress field in the joint, neglecting shear forces. Both of these components are due to the difference in the coefficients of thermal expansion of the joint materials. The first component is the compressive stress in the ceramic. This is mainly caused by the radial force applied by the structural alloy which is transmitted through the braze and interlayer to the ceramic, this is analogous to the "shrink-fit". The second component is the tensile stress in the ceramic. This tensile stress is mainly caused by the metal components of the joint shrinking in a direction parallel to the long axis of the joint. The interlayer controls the largest part of this tensile stress because of its close proximity to the ceramic. The nature of this axial shortening is dramatically shown by the deformed geometry in Figure 2.4. The Ni interlayer shrunk in the axial direction due to its much higher coefficient of thermal expansion as compared to Si_3N_4 .

The mechanical properties of the joint materials play an important part in determining the stresses in the joint. The basic effects of materials' properties on the stress in the joint are discussed below and summarized in Tables 2.2 and 2.3. Figure 2.7 shows that the braze layer supports the majority of the deformation. The flow stress of the braze layer plays a key role in transferring the stresses from the metal components to the ceramic. However, doubling the thickness of the braze layer between the ceramic and interlayer (from 0.005 cm to 0.010 cm) had a negligible effect on the magnitude of the stresses in the ceramic, the compressive stress decreased by 5% while the tensile stress increased by 5%. Doubling the thickness of the braze layer between the interlayer and the metal alloy (from 0.005 cm to 0.010 cm) had a similar effect on the stresses in the ceramic, the tensile stress increased by 5%

while the compressive stress was unchanged. The thicknesses of both braze layers were 0.005 cm at 20°C before brazing but became, 0.020 cm (between the ceramic and interlayer) and 0.0033 cm (between interlayer and structural alloy) at the brazing temperature 1180°C. The thickness of these braze layers changed due to the different coefficient of thermal expansion between the ceramic and metal components of the joint.

Table 2.2. Effects of braze alloy properties on the maximum tensile stress and maximum compressive stress in the ceramic. Properties of the Au-5Pd-2Ni braze alloy (APN) were used as the reference level for comparison with the associated properties of a nickel aluminide (NA). Young's modulus (E), coefficient of thermal expansion (CTE), and σ is the strain-strain relationship. Stresses at the centroid of the elements were used for comparison.

Property of Braze Alloy	Maximum Tensile Stress in Ceramic	Maximum Compressive Stress in Ceramic
Low Young's Modulus (E=APN, CTE=APN, σ =APN)	reference	reference
High Young's Modulus (E=NA, CTE=APN, σ =APN)	small increase (15%)	small increase (20%)
Low CTE (E=APN, CTE=Mo, σ =APN)	small increase (20%)	moderate increase (40%)
High CTE (E=APN, CTE=APN, σ =APN)	reference	reference
Low Flow Strength (E=APN, CTE=APN, σ =APN)	reference	reference
High Flow Strength (E=APN, CTE=APN, σ =NA)	very large increase (440%)	very large increase (125%)

Table 2.3. The effects of interlayer properties of the maximum tensile stress and maximum compressive stress in the ceramic. Properties of Ni were used as the reference level for comparison with the associated properties of Mo. Young's modulus (E), coefficient of thermal expansion (CTE), and σ is the strain-strain relationship. Stresses at the centroid of the elements were used for comparison.

Property of Interlayer	Maximum Tensile Stress in Ceramic	Maximum Compressive Stress in Ceramic
Low Young's Modulus (E=Ni, CTE=Ni, σ =Ni)	reference	reference
High Young's Modulus (E=Mo, CTE=Ni, σ =Ni)	small increase (10%)	no change
Low CTE (E=Ni, CTE=Mo, σ =Ni)	moderate increase (25%)	very large increase (230%)
High CTE (E=Ni, CTE=Ni, σ =Ni)	reference	reference
Low Flow Strength (E=Ni, CTE=Ni, σ =Ni)	reference	reference
High Flow Strength (E=Ni, CTE=Ni, σ =Mo)	large increase (65%)	very large increase (285%)

Figure 2.4. Contour plot of the maximum principal stress in the Si₃N₄/Ni/Incoloy 909 joint at 20°C. The stresses are superimposed on the deformed geometry. The deformations are exaggerated to show the detail. The stress scale reads from 310 MPa to -140 MPa in steps of 30 MPa.

Figure 2.5. Contour plot of the maximum principal stress in the $\text{Si}_3\text{N}_4/\text{Mo}/\text{Inccloy 909}$ joint at 20°C . The stresses are superimposed on the deformed geometry. The deformations are exaggerated to show the detail. The stress scale reads from 1100 MPa to -775 MPa in steps of 125 MPa.

Figure 2.6. Contour plot of the maximum principal stress in the $\text{Si}_3\text{N}_4/\text{Mo}/\text{Inconel 718}$ joint at 20°C . The stresses are superimposed on the deformed geometry. The deformations are exaggerated to show the detail. The stress scale reads from 1500 MPa to -750 MPa in steps of 150 MPa.

Figure 2.7. Total plastic strain in the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ joint at 20°C . The two braze layers support the largest plastic strains. The plastic strain plots for the $\text{Si}_3\text{N}_4/\text{Mo}/\text{Incoloy 909}$ and $\text{Si}_3\text{N}_4/\text{Mo}/\text{Inconel 718}$ joints were similar.

The Young's modulus of the braze alloy had a small effect on the stresses in the ceramic, similar to the effect of the Young's modulus of the interlayer. The thermal expansion coefficient of the braze alloy had a moderate effect on the stresses in the Si_3N_4 ceramic. The reason for the stress increase in the ceramic with a decrease in coefficient of thermal expansion is described below in conjunction with the interlayer properties.

The elastic and thermal properties of the interlayer strongly effect the magnitude of the stresses in the ceramic. At 20°C the maximum compressive stress in the ceramic was 100 MPa for the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ joint and 700 MPa for the $\text{Si}_3\text{N}_4/\text{Mo}/\text{Incoloy 909}$ joint. The high compressive stress in the joint with the Mo interlayer was attributed to the high flow stress and low coefficient thermal expansion (CTE) of Mo.

In a joint with a high CTE interlayer such as Ni, the interlayer and structural alloy contract during cooling from the brazing cycle, and there will be little interference between them. The main source of stress in the ceramic will stem from the interlayer, which in the case of Ni has a low flow stress. However, in a joint with a low CTE interlayer, the interlayer does not contract as much as the structural alloy on cooling from the brazing cycle. The contraction of the high flow stress structural alloy is opposed by the interlayer and this interference stress will be transferred to the ceramic. The deformed geometry shown in Figure 2.4 gives more insight into the effect of the interlayer CTE on the stress level in the ceramic. The deformed geometry for a Mo interlayer is very similar to the deformed geometry of a ductile interlayer with a low CTE. The structural alloy is more compliant at the top of the joint than at the bottom, this results in a hinging motion of the structural alloy. This hinging motion is shown schematically in Figure 2.8. The top of the interlayer is pulled away from the ceramic and the radial stress (shrink-fit stress) at the top of the interlayer becomes tensile in sign. The tensile radial force induces a tensile force in the ceramic through the Poisson effect. This induced tensile force is added to the tensile force in the ceramic due to the axial shrinkage of the interlayer and structural alloy. These 2 tensile forces are additive and result in the increase in tensile force in the ceramic listed in Table III. It should be noted that this behavior is expected to be valid only for cup joint geometries.

Comparing Figures 2.5 and 2.6 for joints with Mo interlayers and Incoloy 909 and Inconel 718, respectively, indicates that a high expansion,

high flow stress structural alloy (Inconel 718) increased stresses in the ceramic. However, it is interesting to note that while the compressive stress increased by 25% (from -700 MPa to -860 MPa) while the tensile stress only increased by 5% (from 220 MPa to 230 MPa). The properties of the structural alloy contribute mostly to the radial stress (shrink-fit) and only slightly to the axial forces.

Figure 2.8. The structural alloy is more compliant at the top of the joint than at the bottom, this results in a hinging motion of the structural alloy.

2.7 Final Joint Geometry

Tables 2.4 and 2.5 list the testing results from the brazed joints tested Phase I. These results are repeated here because the modelling in the following sections was based on these results.

Table 2.4. Results of the torsion tests of $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ brazed joints.

Test Temperature	Torque (N-m)	Shear Strength (MPa)	Bending Moment (N-m)	Rotation	Fracture Mode
25°C	61	151	7	2.22°	Broke in ceramic
	97	242	14	3.52°	"
	53	132	11	1.98°	"
	30	75	13	1.15°	"
	60	148	10	1.05°	"
	49	121	23	2.55°	"
	64	158	14	3.05°	"
	90	222	12	4.3°	"
	45	111	2	2.05°	"
650°C	4.6	11.5	---	---	Slipped; ceramic did not break
	1.6	3.9	---	0.10°	"
	7.0	17.4	---	---	"
	4.9	12.1	---	0.21°	"

Table 2.5. Results of the torsion tests of Si₃N₄/Mo/Inconel 718 brazed joints.

Test Temperature	Torque (N-m)	Shear Strength (MPa)	Bending Moment (N-m)	Rotation	Fracture Mode
25°C	12	30	25	0.38°	Broke in ceramic
	40	98	13	1.19°	"
	23	56	17	0.70°	"
	51	127	16	0.80°	"
	41	101	16	0.64°	"
950°C	0.34	0.84	---	---	Slipped; ceramic did not break

Table 2.6. Results of the 500°C torsion tests of Si₃N₄/Ni/Incoloy 909 brazed joints. All of these joints were fatigued tested at room-temperature before testing at 500°C.

Torque (N-m)	Shear Strength (MPa)	Previous Fatigue Test Conditions			Fracture Mode
		T _{min} (N-m)	T _{max} (N-m)	# cycles	
49.7	123	3.95-20.9	20.9	10 ³	slipped
24.8	61	"	"	10 ⁶	broke in ceramic
15.3	38	6.0	31.7	10 ⁶	slipped

2.7.1 Effect of Shrink Fit

For joints that failed in the joint region, the joint strength was broken down into 2 main parts. The first was the intrinsic strength of the metallurgical bonding across the ceramic/metal interface and the second was the shrink-fit force which was due to the compressive forces exerted on the ceramic by the surrounding metal. Of these two factors the shrink-fit was believed to be the most temperature sensitive. However, at high-temperatures the temperature dependant properties of the joint materials also played a role in the joint strength.

Tables 2.4 and 2.5 showed that the joints were very weak at 650°C and 950°C. It was uncertain whether the low joint strength was due to a weakening of the braze alloy or due to a reduction in the shrink-fit due to the elevate temperature testing. Therefore, the amount of shrink-in (radial stress) in the final joint geometry was calculated by finite element analysis.

The radial stresses were measured from the centroid of the 12 ceramic finite eleme at the ceramic/metal interface. In Figure 2.9, these radial stresses were plotted as a function of distance along the ceramic/metal interface, for several temperatures. The radial stress distribution in the ceramic changed and became very complicated at temperatures above 200°C.

Figure 2.9. Centroid stresses in the ceramic at the ceramic/interlayer interface as a function of temperature calculated by finite element analysis for a $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ joint. The joint was initially cooled down to 20°C from the brazing temperature then reheated to the different temperatures.

The stresses in Figure 2.9 were used to estimate the loss in joint strength as a result of an increase in temperature and the resultant decrease in shrink-fit. For a shrink-fit joint, the torque (T) the joint can withstand without slipping was approximated by the following equation:

$$T = \mu r A \sum_{i=1}^n P_i \quad \text{Eq. 2.1}$$

where μ was the coefficient of friction between the ceramic and metal (~ 0.1), 'r' was the radius of the ceramic (6.35 mm), 'A' was the area of contact which was taken as the area/element in the FEA mesh ($1.7 \times 10^{-5} \text{ m}^2$) times the number of elements (n) with a compressive stress, and P_i was the radial

compressive stress in element 'i' (σ_{rr}). Only compressive stresses were included in this analysis. The radial stresses along the ceramic/metal interface are shown in Figure 2.9. The calculated values of the torque due to the shrink-fit stresses were 122 N-m at 20°C, 25 N-m at 500°C and 1.8 N-m at 650°C, corresponding to shear strengths of 304 MPa, 62 MPa, and 4 MPa, respectively. These torque values are for a shrink-fit joint and are only valid for joints that slipped at the ceramic/metal interface and did not break in the ceramic or fail by deformation of any of the metallic components of the joint.

The total strength of a joint can be thought of as being comprised of two components; the shrink-fit, and the metallurgical bond. The metallurgical bond component is the chemical bonding and mechanical interlocking at the ceramic/metal interface, in this case between the Ti-coating and silicon nitride. The total joint strength is defined as;

$$\text{total joint strength} = \text{shrink-fit} + \text{metallurgical bond} \quad \text{Eq. 2.2}$$

where the contribution of the metallurgical bond to the joint strength was estimated from a previous study to be constant at 85 MPa for the temperature range studied (see Table V [8]).

For the joints tested at room-temperature the total joint strength is approximately 304 MPa + 85 MPa = 389 MPa. This strength is significantly higher than the measured joints strengths, Table 2.4, which is supported by the fact that at room-temperature the ceramic broke and the joint did not slip, i.e., the joint was stronger than the ceramic. At 500°C the total joint strength is approximately 62 MPa + 85 MPa = 147 MPa, which compares favorably with the experimental value of 123 MPa after 1000 cycles of fatigue testing, Table 2.6. At 650°C the total joint strength is approximately 4 MPa + 85 MPa = 89 MPa. This value is significantly higher than the experimental value of approximately 10 MPa, Table 2.4. This discrepancy is attributed to a weakening of the braze alloy at 650°C, indicating that at the higher temperatures the joint strength is controlled by the strength of the braze alloy.

The mechanical properties of the braze alloy at elevated temperatures were expected to play an important role in determining the creep behavior of this joint. However, the creep requirements for this joint were not well defined and were not studied.

2.7.2 Comparison Between FEA Predictions and Experimental Results

Finite element analysis was used to predict the residual stress for each joint component. The joints were assumed to have perfect bonding at every interface, the presence of voids in the braze was ignored. Further, the influences of metallurgical reactions due to processing was not accounted for in the analysis. Bulk properties were used for each material in the joint. Also, significant alloying was seen between the braze, interlayer, and structural alloys. This alloying caused a change in the braze alloy's composition and hence in its properties (the most important was the flow stress). Such effects were not accounted for in the finite element analysis.

The comparison between predictions and experimental results were made with (1) a maximum principal stress approach and (2) a probabilistic approach to joint failure. The first approach was based on the assumption that the joint failure was controlled only by the magnitude of the tensile principal stress. That is, failure of the joint was assumed to occur at the maximum tensile stress point regardless of the directionality of the stress as long as the sum of the residual stress and applied stress exceeds the strength of the ceramic. For the probabilistic approach, the NASA CARES program [2,3] was used to calculate the probability of survival (P_s) of the joints and the result was presented in the form of a probability plot. In this approach, the predicted strength value was obtained by superimposing the distribution of an applied torque and the calculated residual stress.

2.7.2.1 Maximum Principal Stress Approach

This approach had the advantage of being mathematically simple. The situations with and without residual stress were represented by the properties of the brazed joint and of the unbrazed ceramic sample, respectively. For ceramic-metal joints, the fracture strength of the joint was expressed as a function of residual stress and applied stress:

$$\tau_{\text{fracture}} = f(\tau_{\text{residual}}, \tau_{\text{applied}}), \quad \text{Eq. 2.3}$$

where τ_{fracture} was the measured fracture shear stress of the unbrazed

ceramic, τ_{residual} was calculated by finite element analysis, and τ_{applied} was the measured fracture shear stress of the brazed joint.

For this analysis, cylindrical coordinate system was employed for the ceramic-metal joint. The stresses determined by finite element analysis, σ_{zz} and $\sigma_{\theta\theta}$, were the maximum principal stresses taken from the free surface in the vicinity of the ceramic-metal joint (i.e., σ_1 and σ_3 respectively). The stress σ_{rr} was zero on the free surface, resulting in a plane stress condition. These stresses were converted into a maximum shearing stress for comparison with the measured joint strength by a Mohr's circle analysis.

Substituting for the residual stress quantities, the relationship between the residual stress in the joint and the strength of the joint, $\tau_{\text{predicted}}$, becomes

$$\sigma_{\text{max}} = \frac{\sigma_{zz} + \sigma_{\theta\theta}}{2} + \sqrt{\left(\frac{\sigma_{zz} - \sigma_{\theta\theta}}{2}\right)^2 + (\tau_{\text{predicted}})^2}, \quad \text{Eq. 2.4}$$

where σ_{max} equaled τ_{max} in a torsion test based on the following relationship:

$$\tau_{\text{max}} = \frac{\sigma_{11} - \sigma_{33}}{2} = \frac{\sigma_{11} - (-\sigma_{11})}{2} = \sigma_{11} = \sigma_{\text{max}}, \quad \text{Eq. 2.5}$$

and τ_{max} was the measured shear strength of the unbrazed ceramic i.e., τ_{fracture} , from a torsion test. For real, nonideal samples, Equation 2.4 becomes

$$\tau_{\text{fracture}} = \frac{\sigma_{zz} + \sigma_{\theta\theta}}{2} + \sqrt{\left(\frac{\sigma_{zz} - \sigma_{\theta\theta}}{2}\right)^2 + (\tau_{\text{applied}} + \Delta\tau)^2}, \quad \text{Eq. 2.6}$$

where τ_{applied} was the shear strength for fracture measured from brazed samples. $\Delta\tau$ denoted the difference between $\tau_{\text{predicted}}$ and τ_{applied} due to processing, testing errors, and the probability factor for cracking in the ceramic material etc.. A comparison was be made between the predicted values, $\tau_{\text{predicted}}$, in Equation 2.4 and the measured values, τ_{applied} , in Equation 2.6.

The values of σ_{zz} and $\sigma_{\theta\theta}$ at 20, 500 and 650°C from the finite element analysis study are listed in Table 2.7. These values were taken from the centroid of the mesh element on the free surface of the ceramic immediately above the joint. This element exhibited the most severe stress state in the joint region and was therefore used to predict the joint strength. This element did not represent the exact stress conditions, i.e., plane stress, on the surface. However, these values were taken as the principal stresses since the stress values of the third component, σ_{rr} , were significantly smaller than those of σ_{zz} and $\sigma_{\theta\theta}$. The magnitude of σ_{rr} was typically 1% of σ_{zz} .

Table 2.7. Comparison between finite element analysis (FEA) predictions and experimental joint strengths of $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ joints. $\tau_{\text{predicted}}$ was calculated with Equation 2.4 and a τ_{max} of 593 MPa. $\tau_{\text{corrected}} = 1.7 \times (\tau_{\text{predicted}})$.

Test Temperature	FEA Prediction σ_{zz} $\sigma_{\theta\theta}$ (MPa)		$\tau_{\text{predicted}}$	τ_{measured}	$\tau_{\text{corrected}}$	Failure Mode
			(MPa)			
20°C	131	12	513	75-242	127-413	Broke in ceramic
500°C	-60	-19	628	38-123*	65-209	Slipped or Broke in ceramic
650°C	-51	-16.8	622	3.9-17.4	6.6-30	Slipped

* These joints were fatigued at 20°C for 10^3 or 10^6 cycles prior to testing.

Using Equation 2.4 and τ_{max} , 593 MPa (the strength of PY6), the calculated value of $\tau_{\text{predicted}}$ at room-temperature was 513 MPa while the measured values from the torsion tests were in the range of 75-242 MPa. The measured values became 127-413 MPa when the stress concentration factor (K_t

= 1.7) associated with the change in diameter at the joint was accounted for. The measured values are 25-80% of the values predicted by finite element analysis.

Selection of the stress concentration factor depended on the radius of the fillet formed by the braze alloy. A fillet radius of 1 mm was selected, however the fillet radius varied over the joint area. The stress concentration factor was estimated from Rourk [9].

At 500°C and 650°C, σ_{zz} and $\sigma_{\theta\theta}$ became compressive and Equation 2.4 resulted in a significantly higher $\tau_{\text{predicted}}$, 628 and 622 MPa for 500°C and 650°C, respectively. These compressive forces resulted in the ceramic in the joint region being strengthened with respect to the ceramic outside of the joint region. Therefore, at these elevated temperatures the ceramic in the joint region was not predicted to fail, failure was predicted to occur in the ceramic away from the joint region.

Joints were fatigued 10^3 and 10^6 cycles at a torque amplitude of 3.95-20.9 N-m. These joints were subsequently torqued to failure at 500°C, one joint failed in the ceramic near the joint region while the other joints slipped at the ceramic-metal interface (see Table 2.6). At 650°C, all of the joints slipped at the ceramic-metal interface. The finite element analysis values of σ_{zz} and $\sigma_{\theta\theta}$ at 500°C and 650°C were obtained from the joint model that was cooled down from the brazing temperature to room-temperature and heated back up to 500°C and 650°C, respectively. The values of τ_{measured} , 38-123 MPa and 3.9-17.4 MPa, represent the strength of the weakest component, i.e., the interface region, at 500°C and 650°C, respectively. Since the failure theory only dealt with ceramic failure, the strength predictions at 500°C and 650°C were invalid because failure occurred in the metal components and not in the ceramic.

2.7.2.2 Ceramic-Failure Model for Bimaterial Systems

A new ceramic failure theory was developed for use in a bimaterial system. The essential feature of this theory is that the plasticity of the metal member of the joint causes a reduction in the stresses in the ceramic member. This reduction in stress is a consequence of the metal member plastically deforming due to the stress field in the ceramic near the bimaterial interface. The stress reduction is expressed by a series of modification factors listed below;

$$\sigma_{rr}^{eff} = \frac{\sigma_{rr}}{\sqrt{f_c}} \quad \text{Eq. 2.7a}$$

$$\sigma_{r\theta}^{eff} = \frac{\sqrt{\lambda} \sigma_{r\theta}}{\sqrt{f_c}} \quad \text{Eq. 2.7b}$$

$$\sigma_{rz}^{eff} = \frac{\sqrt{\lambda} \sigma_{rz}}{\sqrt{f_c}} \quad \text{Eq. 2.7c}$$

$$\sigma_{\theta\theta}^{eff} = \sigma_{\theta\theta} \quad \text{Eq. 2.7d}$$

$$\sigma_{z\theta}^{eff} = \sigma_{z\theta} \quad \text{Eq. 2.7e}$$

$$\sigma_{zz}^{eff} = \sigma_{zz} \quad \text{Eq. 2.7f}$$

where "r", "z", and "θ" have their usual meanings in a cylindrical coordinate system, $\lambda = 0.3$, and the σ values on the right hand side of the equations are calculated from the FEA code.

$$f_c = \frac{G^\infty}{G} \quad \text{Eq. 2.8a}$$

$$f_c = \exp \left(a \frac{K_{Ic}}{\sigma_0 \sqrt{h}} + b \frac{K_{Ic} \sqrt{t}}{\sigma_0 \sqrt{h}} + c \frac{K_{Ic}^2}{\sigma_0^2 \sqrt{t h}} \right) \quad \text{Eq. 2.8b}$$

where G^∞ is the total energy release rate due to far field loading, G is the actual energy release rate calculated at the crack tip, "a" = 0.0837, "b" = 0.0447, "c" = 0.711, "t" is the thickness of the braze layer, "h" is the distance from the braze layer to the location under consideration in the ceramic, and K_{Ic} is the fracture toughness of the ceramic. The dependance of f_c on "h" in Equation 2.8 results in the effective stresses in Equation 2.7 being significantly modified

only when "h" is small, i.e., near the braze layer. Once the effective stresses (σ^{eff}) are calculated they are used in the CARES code with which ever failure theory is selected. The effect of incorporating the metal plasticity is to reduce the stresses in the ceramic and increase the probability of survival, P_s . The underlying theory and detailed derivation of the above equations are listed in Appendix B.

2.7.2.3 Probabilistic Approach

For this approach, an applied torque was superimposed with the calculated residual stress field as a shear stress. Figure 2.10 shows the torque distribution used to determine the shear stress. A stress concentration due to the change in cross-section at the joint was included as before. The stress concentration was 1.7 at the top of the joint and tapered off to 1.0 at a distance of one ceramic diameter away from the joint. The fraction of torque carried by the ceramic and metal components in the joint region was calculated based on the material properties and was also factored into the torque distribution shown in Figure 2.10. Once the torque was added to the residual stress field, the combined stress field was used as input to the CARES probabilistic failure computer code to obtain the probability of survival for the joint at the applied torque level. This same procedure was performed at several different torques and the probability of failure (P_f) values were calculated as a function of applied torque. This probabilistic approach to joint strength, unlike the maximum principal stress approach, provided a method to estimate the strength distribution of a ceramic-metal brazed joint and can be used as a design aid.

Two failure theories were used to predict the behavior of the ceramic-metal joints for comparison. These were; 1) Shetty criterion, with $\bar{C} = 0.82$, a penny-shaped crack, and a shear-insensitive Batdorf crack density coefficient, and 2) coplanar strain energy release rate, with a Griffith crack, and a shear-sensitive Batdorf crack density coefficient. For these failure theories the Weibull modulus (m_v), the normalized Weibull scale parameter (τ_{0v}), and the Batdorf crack density coefficient (k_{Bv}) were calculated from 4-point bend tests of the silicon nitride material used in the ceramic-metal brazed joints. Specimen measuring 2.54 mm x 1.27 mm x 25.4 mm were used with an inner and outer test span of 10.16 mm and 22.86 mm for the 4-point bend tests, the tensile surface of the test bars were polished to 0.1 μm . Twenty-

four samples were tested and 1 outlier was detected in the data. The data is shown in Figure 2.11.

A Weibull modulus (m_v) of 21.2 and a normalized Weibull scale parameter ($\sigma_0 v$) of $369.8 \text{ MPa(m)}^{3/21.2}$ were used for both failure theories. A shear-insensitive Batdorf crack density coefficient of 43.4 and a shear-sensitive Batdorf crack density coefficient of 22.2 were used for the Shetty and coplanar strain energy release rate theories, respectively. The maximum likelihood method was used to fit the experimental data. The constants described above were required as input variables by the CARES and are further described by Nemeth et al. [2].

The failure theory described in section 2.1 was also used to predict the strength of these joints. This theory was used to modify the stresses predicted by FEA prior to using CARES to calculate P_s . The predictions given using the theory in section 2.1 are termed "modified".

Figure 2.12 and 2.13 show predicted and experimental results of torsion testing of the brazed joints. The predicted strength distributions surrounded the experimental values and intermediate values of P_f , where $P_f = 1 - P_s$. However, at the low and high regions of the probability distributions the predictions deviated from the experimentally measured strengths. The use of the theory in section 2.1 resulted in an increase in predicted strength of 10 - 20%. This increase in the predicted strength was expected since the theory in section 2.1 results in a reduction in the effective stress due to plastic deformation of the braze layer.

The predicted probability distributions were much steeper (higher Weibull modulus) than the experimental probability distribution. The Weibull modulus decreased from 21.2 for the unbrazed ceramic to ~ 4 for the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ brazed joints. A similar decrease in Weibull modulus was seen in shear testing of ceramic-metal lap joints [8] and in 4-point bend tests of ceramic-metal butt joints [10]. The cause of the difference in the slopes (Weibull modulus) of the probability distributions was unknown but may be due to subcritical crack growth in the ceramic.

Figure 2.10. Plot of the torque distribution in the ceramic portion of the brazed joint. Torque values are normalized to 1.0, i.e., the applied torque.

Figure 2.11. Weibull plot of 4-point bend test data of PY6. The data point represented by the open circle was considered by CARES as an outlier and was not used to determine the material parameters.

Figure 2.12. Predicted and experimental values of the brazed joint strength measured in torsion ($\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$). Two failure theories were used - 1) Shetty criterion with $\bar{C} = 0.82$, penny-shaped crack, and a shear-insensitive Batdorf crack density and 2) Coplanar strain energy release rate, Griffith crack, and a shear-sensitive Batdorf crack density.

Figure 2.13. Predicted and experimental values of the brazed joint strength measured in torsion ($\text{Si}_3\text{N}_4/\text{Mo}/\text{Inconel 718}$). Two failure theories were used - 1) Shetty criterion with $\bar{C} = 0.82$, penny-shaped crack, and a shear-insensitive Batdorf crack density and 2) Coplanar strain energy release rate, Griffith crack, and a shear-sensitive Batdorf crack density.

2.7.2.4 Comparison of Prediction Methods

The difference between the predicted and the experimental strengths of the joints in both approaches (maximum principal stress and probabilistic) was mainly attributed to differences in the ideal joints modelled and the real joints tested. The joints were expected to have some unbonded regions. Nonuniform bonding resulted in the loss of the axial symmetry in the stress state. This resulted in the loss of the plane stress condition at the point of maximum residual stress. The triaxial stress state at the surface, caused by the unbonded area in the joint, reduced the effective stress for the failure of the

ceramic. This will increase the difference between experimental values and finite element analysis predicted values of the joint strength. Also, during the brazing process, a change in the compositions and properties of the joint materials contributes to error in the finite element predictions. The presence of molten braze enhances the dissolution of other joint components, causing new phases to form and compositional changes of the braze alloy. These will lead to changes in the mechanical properties of the joint.

The maximum principal stress criterion assumed that ceramic survival or failure was a function of stress only and predicted a single strength for the joint. This implied that the failure of the joint should always coincide with the point of maximum residual stress. Overall results showed that the maximum residual stress provided a good estimate of the location of probable fracture of the joint [1]. However, the probabilistic approach for the ceramic-metal brazed joints provided a method to compare the performance of joints with different geometries; and to incorporate the statistics of ceramic failure into the prediction method. The probabilistic approach to failure prediction also predicted the performance of the joint as a function of stress, and resulted in a range of joint strengths.

In this study, the failure theories employed in the strength prediction did not adequately treat ceramic failure resulted from compressive stresses. The contribution of compressive stresses to ceramic failure is expected to be significant due to the high level of compressive stresses induced in the ceramic by the thermal expansion mismatch between the joint materials.

3. JOINT MATERIALS DEVELOPMENT

3.1 Structural Materials for Jointing

3.1.1 PY6 Silicon Nitride

GTE PY6 silicon nitride was chosen as the structural ceramic for Phase II of the program. It is known that PY6 has superior high-temperature strength and compatibility with reactive coating materials compared to other silicon nitrides [11]. Excellent high-temperature performance of this material is attributed to the presence of crystalline grain boundary phases. The coefficient of thermal expansion of PY6 is reported $3.4 - 4.0 \times 10^{-6}/^{\circ}\text{C}$ in the temperature range of $25 - 1000^{\circ}\text{C}$. PY6 was sintered first and further densified by HIPing at 1825°C using a glass encapsulation technique. The products are in the form of rectangular bars or cylindrical rods. All sample surfaces were polished down to $0.03 \mu\text{m}$ with alumina powder for experiments. The rod samples were machined and polished at Carbide Specialty in Waltham MA. Figure 3.1 shows the Weibull plot of PY6 used for Phase II along with previous materials used for Phase I.

Figure 3.1. Weibull plot of PY6 silicon nitride for Phase I and II

3.1.2 Incoloy 909

Incoloy 909 has become a common choice as a structural alloy for ceramic-metal joints because it has a low coefficient of thermal expansion as well as high strength. This precipitation-hardened Fe-based alloy (Fe-38Ni-13Co-5Nb-1.5Ti) has been used for gas-turbine casing, shrouds, vanes and shafts. In the brazing of Incoloy 909 to ceramics, there has been a concern over the effect of the high-temperature brazing process ($>1000^{\circ}\text{C}$) on the mechanical properties of Incoloy 909. Two points were studied; (1) the extent of degradation in the mechanical properties of Incoloy 909 after heat treatment at a high-temperature ($>1000^{\circ}\text{C}$) and (2) the possibility of the strength recovery of this alloy during the brazing process. In this study, the effects of high-temperature brazing on the mechanical properties of Incoloy 909 were evaluated after various heat treatments, and a post-heat treatment procedure was demonstrated to restore the strength of Incoloy 909.

According to the manufacturer, Huntington Alloys, the alloy is normally homogenized at 1200°C (2200°F), solution heat treated at 980°C (1800°F), and double aged at 750°C (1375°F) for 4 hours and at 620°C (1150°F) for 4 hours to strengthen it. Therefore, the use of Incoloy 909 is limited by the solution heat treatment (SHT) temperature. This alloy is also known to suffer oxygen embrittlement at high-temperatures due to the lack of Cr in the alloy. In aircraft brazing applications (1050°C brazing), the manufacturer recommends overaging treatment along with solution treatment at 1050°C . Table 3.1 lists the manufacturer's specifications for the room-temperature and 650°C tensile properties of Incoloy 909 prepared by the regular heat treatment and overaging treatment.

Table 3.1 Tensile properties of Incoloy 909 - manufacturer's specifications.
SHT = solution heat treatment. AT = ageing treatment.

Heat Treatment Conditions	Test Temp (°C)	UTS (ksi)	YS (ksi)	% Elongation
SHT - 980°C (1 hr), AT - 750°C (4 hr) and 620°C (4 hr)	RT	180	130	15
	650	130	100	15
SHT - 1050°C (1 hr), AT - 770°C (8 hr) and 620°C (4 hr)	RT	115-120	100	10
	650	125	90-95	10-15

In Phase I of the ceramic-metal joining program, PY6 silicon nitride was joined to Incoloy 909 via brazing at 1180°C with an Au-Pd-Ni alloy. For Phase II, new brazing alloys which have good creep strength at 600-650°C are being developed. As the application temperature of the joints increases, it is mandatory for a new braze alloy to have a high melting point.

For the experiments, Incoloy 909 was solution heat treated in vacuum at 1200°C for 30 minutes to exaggerate the brazing condition. The time at the brazing temperature is in the range of 10-30 minutes. In addition, the solution treated alloys were aged in the temperature range of 600-1000°C for 1-24 hours in air. Table 3.2 shows the results of hardness tests (R_B) after various aging heat treatments. The SHT at 1200°C for 30 minutes resulted in a reduction of R_B from 102 to 89 by the resolution of precipitates. Subsequent aging treatments resulted in gradual gains in hardness, depending on aging temperature and time. Of these aging temperatures, a quick response was noted from aging at 600°C.

Table 3.2. Rockwell B (R_B) hardness of Incoloy 909 after various heat treatments.

Heat Treatment Conditions	Time for Aging Treatment (Hrs.)							
	0	1	2	3	4	8	12	24
As-received	102							
SHT at 1200°C (10 min) in vac.	92							
SHT at 1200°C (30 min.) in vac.	89							
SHT at 1200°C (30 min.) in vac., AT at 600°C in air	---	92	95	98	100	100	95(?)	104
SHT at 1200°C (30 min.) in vac., AT at 800°C in air	---	89	90	89	91	107	108	112
SHT at 1200°C (30 min.) in vac., AT at 1000°C in air	---	87	87	87	86	---	---	---

Based on the hardness results, representative heat treatment conditions (600 and 800°C) were selected for Incoloy 909, and the tensile properties were measured using substandard sized samples of 0.25" diameter. The results are listed in Table 3.3. Compared to the manufacturer's strength specifications, the properties of as-received Incoloy 909 were significantly lower, while the elongation values were higher. The solution heat treatment at 1200°C for 30 minutes reduced the tensile strength by 25% (from 132 ksi to 98.5 ksi) without a loss of ductility. Processing in vacuum (10^{-5} torr) prevented the alloy's embrittlement and prevented a loss in ductility. Aging at 600°C for 4-24 hours resulted in full strength being attained. As expected, aging treatments (AT) in air for 24 hours caused a reduction in ductility. Aging treatment at 800°C seemed to have caused overaging of the alloy as well as noticeable embrittlement. The AT's at 600°C and 800°C were done in air to simulate actual service conditions.

Table 3.3. Tensile properties of Incoloy 909 after various heat treatments.
Average of three tests.

Heat Treatment Conditions	UTS (ksi)	YS (ksi)	% Elongation
As-Received	132	81	52
SHT*	99	52	58
SHT*, AT 600°C (4 hrs.) in air	121	89	48
SHT*, AT 600°C (24 hrs.) in air	139	107	33
SHT*, AT 800°C (4 hrs.) in air	88	51	37
SHT*, AT 800°C (24 hrs.) in air	105	-	11

* SHT (Solution Heat Treatment) was done at 1200°C for 30 min., subsequently followed by AT.

Attempts were made to determine the impact of our current high-temperature brazing at 1180°C for 10 minutes on the mechanical properties of Incoloy 909. Also, a one-step high-temperature brazing process, which could restore the strength of the alloy, was developed. This included SHT at 1200°C for 10 or 30 minutes and subsequent AT at 600°C for 4 hours in vacuum. The results are shown in Table 3.4. High-temperature brazing at 1180°C lowered the tensile strength by 15% and the yield strength by 20% without a loss of ductility, compared to as-received 909. However, the subsequent AT at 600°C for 4 hours after SHT for 10 minutes increased the yield strength of the alloy with some loss of the ductility. Metallography and tensile tests with the alloys showed that high-temperature processing, for example, SHT 1200°C for 30 minutes and AT at 600°C for 4 hours, induced grain growth of the alloy from 250 μm to 350 μm , resulting in softening of the alloys.

Table 3.4. Tensile properties of Incoloy 909 after one-step process.

Heat Treatment Conditions	UTS (ksi)	YS (ksi)	% Elongation
1180°C for 10 min. (typical brazing cycle)	111	65	52
1200°C for 10 min., 600°C for 4 hrs. in vac. (typical brazing & heat treatment)	135	101	44
1200°C for 30 min., 600°C for 4 hrs. in vac. (extended brazing & heat treatment)	117	89	44

Samples were prepared to see the changes in microstructure after aging treatment. TEM specimens were ground then ion-milled. Figures 3.2 and 3.3 show the microstructures of as-received Incoloy 909 and heat treated Incoloy 909, respectively. The as-received sample retained small grain size in the range of 0.5-1 μm without any precipitates in the structure. The absence of precipitates indicates that the solution and aging treatment of this alloy by the manufacturer were not sufficient to induce proper precipitation hardening of this alloy. This explains the discrepancy in the mechanical properties shown in Tables 3.1 and 3.2. The sample which was overaged at 800°C for 4 hours in air after solution treatment at 1200°C for 30 minutes showed needle shaped precipitates (Figure 3.3). Preliminary study showed that this precipitate might be a Ni_3Nb -type phase and that this phase forms on a particular habit plane. The morphology and distribution of the phase seems to be responsible for the tensile strength of the alloy.

Overall results showed that high-temperature brazing at 1200°C lowered the strength of the alloy as the brazing time at the temperature increased. However, it was confirmed that the strength loss caused by high-temperature processing such as brazing could be minimized or avoided when proper post-heat treatment was used. Therefore, the service life of the alloy for 600-650°C application is more related to the intrinsic high-temperature properties of the alloy than to the processing history.

Figure 3.2. TEM micrographs of as-received Incoloy 909.

Figure 3.3. TEM micrographs of heat treated Incoloy 909 - SHT at 1200°C for 30 minutes and aged at 800°C for 4 hours.

3.2 Brazing Alloy Design

3.2.1 Approach for Alloy Development

Most commercial high-temperature brazing alloys were designed for metal-metal joints and matched the compositions and mechanical properties of parent materials as closely as possible. It is expected that the joints can maintain desirable high-temperature strength, hot corrosion resistance, and creep strength. Common brazing alloys for high-temperature applications are Ni-based alloys. There is no brazing alloy designed specifically for ceramic-metal joints for use at high-temperatures. Due to the strict property requirements of brazing alloys for this kind of joints, Au- or Pd-based alloys which contain more than 80% of precious metals in most cases are common alternatives. Alloy compositions were selected to provide high melting points with a small liquid+solid temperature range and without a loss of ductility of the alloys. Some of these alloys, depending on chemical compositions, have provided reasonable bonding and room-temperature performance. However, high-temperature performance in dynamic load bearing conditions was always disappointing due to the nature of precious metals. For 650°C application, new alloys need high-temperature creep strength as well as oxidation resistance.

As shown in previous sections, an FEA (finite element analysis) study indicated that the alloys need to have low yield strength, high ductility, and high ultimate strength at low temperature for optimum bonding performance. High ductility and high toughness values of the alloys are extremely important in producing braze foils by rolling. Many of these requirements are in conflict. Interactions between joining components sometimes cause compatibility problems. There is a limit in brazing temperatures to prevent silicon nitride substrates from decomposing, $\sim 1200^{\circ}\text{C}$.

Figure 3.4 illustrates the procedure for high-temperature brazing alloy development from alloy screening to the testing schedule for evaluating the joints made of new alloys. As mentioned, master alloys will be selected based on the requirements of the joints during brazing and applications. Candidate compositions will be evaluated with wetting tests and hardness tests. Wetting tests were done on the coated ceramic and interlayer materials in an argon atmosphere, and Rockwell B hardnesses were measured. Simple bonding tests were done with coupon samples and braze alloys which showed desirable properties. The most successful compositions will be further modified and

refined by incremental changes in the ratio between individual elements. Final compositions will be cast into a copper mold and rolled into foils.

Optimum brazing conditions were developed for different joint geometries by measuring joint strength as a function of brazing temperature and time at temperature. Lap joints were used for shear testing, whereas cylindrical joints were used to measure high-temperature creep property, room-temperature torsional joint strength, and thermal and mechanical fatigue properties.

The approach was taken with Pd-containing alloys to understand the effect of interactions between alloys and the Ti coating layer on the brazing performance. Two other approaches with Ni-based or Au-based alloys were to develop high-temperature brazing alloys for ceramic-metal joints with emphasis on either (1) brazability of ceramic-metal or (2) high-temperature performance.

Figure 3.4. Procedure for brazing alloy development.

The primary reason for the existence of Pd in the Au-Ni system was to raise the melting points of the alloys without loss of the single-phase microstructure. However, it was found that Pd-containing alloys often resulted in processing difficulties due to the interactions between the alloys and coating materials. Therefore, the effect of alloying elements such as Ni and Pd on the processing and mechanical properties of the joints was studied using commercial Pd-containing braze alloys. The results of this study will be a reference for new alloy development.

Design emphasis was more on high-temperature creep strength than on brazability. Since most Ni-based alloys have high melting points ($>1300^{\circ}\text{C}$) and low ductility values compared to the requirements of joint production, elements such as Au, Cu, Ag (IB), Pd or B, Si, Ge or Sn, and In were added to lower the melting points with a minimum loss of ductility. Solid solution strengthened Ni alloys were preferentially selected over precipitation strengthened Ni alloys which normally contain Ti and Al to form γ' or γ'' in the structure. Solid solution strengthened Ni alloys which have Cr, Fe, Mo, and Nb as strengtheners provide relatively low yield strength with high ductility. It was aimed for the alloys to maintain a solid solution single phase structure without having precipitation hardening phases after additions of alloying element(s).

The design strategy with Au-based alloys is to improve high-temperature creep strength of Au-based alloys by adding elements of high melting points and solid solution formers such as Ni, Cr, Mo, and Fe. Au-Ni alloys were selected as a base composition due to its single-phase microstructure and excellent room-temperature mechanical properties. It was intended for Au-Ni alloys to introduce dual solid solution phases in the microstructure as a result of alloying with Ni, Cr, Fe, and Mo. The dual phases were expected to enhance the resistance to high-temperature deformation. Plans were also made to add Cu to the alloys to reduce the amount of Au.

3.2.2 Candidate Alloy Systems

3.2.2.1 Pd-based alloys

There are several Pd containing braze alloys commercially available. These brazes had Pd as an addition to raise the melting point. Several commercially available Pd containing brazes were evaluated for their high-temperature properties and their ability to successfully joint silicon nitride and metal.

3.2.2.1.1 Tensile Properties of Pd-Containing Brazes

The effect of Pd additions to the mechanical and chemical properties of Au-Ni braze alloys was studied. Six braze alloys were studied: 60Pd-40Ni, 50Au-25Pd-25Ni, 30Au-34Pd-36Ni, 70Au-8Pd-22Ni, 93Au-5Pd-2Ni, and 82Au-18Ni all in wt% (Table 3.5).

The Pd-containing alloys mentioned above are solid solution strengthened, and their application temperatures are usually limited to below 500°C. In order to establish a baseline data for the development of high-temperature creep resistant braze alloys, the mechanical properties of thin foils of these alloys were evaluated; 82Au-18Ni was annealed at 850°C for 5 hours before tensile tests, whereas all other foils were treated at 900°C.

These alloys did not exhibit a particular pattern in strength-composition relationship. However, the highest foil strength (728 MPa yield strength) at room-temperature was observed from the 50Au-25Ni-25Pd alloy which resulted in the poorest brazing performance among the alloys studied. Alloys 82Au-18Ni, 70Au-8Pd-22Ni, 30Au-34Pd-36Ni, 60Pd-40Ni, and 93Au-5Pd-2Ni showed 609, 588, 564, 294, and 88 MPa yield strength, respectively. Additions of Au, up to about 85%, seemed to be effective in providing high foil strength.

This study found that the addition of Pd to the Au-Ni system increases the high-temperature capability of the alloys at the cost of strength, ductility, and, more significantly, brazability. The low strength of 60Pd-40Ni, with an increased modulus of elasticity, indicates the embrittlement of this alloy. Based on this study, it seems that the Au-Pd-Ni system is limited to the capacity obtainable from the Au-Ni system, with up to 10% of Pd addition.

Table 3.5. Liquidus (T_L) and brazing (T_B) temperatures of the braze alloys, along with selected room-temperature mechanical properties of the braze foils, and the room-temperature coefficient of thermal expansion (CTE).

Trade Name of Braze Alloy†	Au Pd Ni (wt. %.)	T_L (°C)	T_B (°C)	E (GPa)	σ_T (MPa)	σ_y (MPa)	CTE ($\times 10^{-6}/^\circ\text{C}$)
Palni	--- 60 40	1238	1290	47.3	373	292	---
Palniro 4	30 34 36	1169	1220	30.0	574	574	15.5
Palniro 1	50 25 25	1121	1170	35.0	735	728	15.5
Palniro 7	70 8 22	1037	1090	42.7	600	585	17.5
Experimental	93 5 2	1082	1180	28.7	109	88	---
Niuro	82 --- 18	950	1000	49.0	760	606	17.5

† GTE WESGO, Belmont, CA.

3.2.2.1.2 Strengths of Joints Brazed with Pd-Containing Brazes

Coupons were brazed using the above alloys, Ti-coated SNW1000 Si_3N_4 and Ni. SNW1000 Si_3N_4 contained 13 wt% Y_2O_3 and 2 wt% Al_2O_3 as sintering aids. Ten coupons were brazed with each braze. Five coupons were shear tested at room-temperature and 500°C in an argon atmosphere, for each braze.

The high Pd brazes, 60Pd-40Ni, 50Au-25Pd-25Ni, and 30Au-34Pd-36Ni, had very low shear strength values; all were below 35 MPa (Tables 3.6 and 3.7). Most of the coupons brazed with the high Pd-containing brazes broke during handling. Of the 30 samples made with these brazes only 12 survived handling and were tested. All of the samples brazed with 50Au-25Pd-25Pd braze were cracked in the Si_3N_4 on cooling and could not be tested. The cracks often propagated all the way through the Si_3N_4 .

The joints that cracked on cooling from the brazing temperature had either a high yield strength braze or were high-temperature brazes and therefore were subjected to large thermal residual stresses. The coefficients of thermal expansion of these brazes are not known as a function of temperature, but they are expected to be approximately $15 \times 10^{-6}/^\circ\text{C}$ near room-temperature.

There was not a significant difference in the fracture mode of any of the samples, Table 3.8, except for the 60Pd-40Ni brazed coupons tested at 500°C .

Most of the samples fractured in the ceramic (at a depth of approximately 1-2 mm), with the crack propagating parallel to the interface. The joints with the 60Pd-40Ni braze had approximately 30% unbonded area. The fracture surface of the 50Au-25Pd-25Ni brazed sample was typical of the fracture surfaces from the four other braze alloys.

Table 3.6. Results from room-temperature and 500°C shear tests between Ni and Ti-coated Si₃N₄.

Braze Alloy Au Pd Ni (wt. %)			Failure Load (kN)											
			25°C					500°C						
			Sample #					Sample #						
			1	2	3	4	5	Avg.	1	2	3	4	5	Avg.
---	60	40	1.6	*	*	*	*	1.6	2.6	4.4	3.2	0.8	*	2.8
30	34	36	0.2	0.5	*	*	*	0.4	3.3	*	*	*	*	3.3
50	25	25	*	*	*	*	*	*	*	*	*	*	*	*
70	8	22	9.9	7.1	6.4	7.6	18.5	12.0	10.5	11.6	9.2	9.9	11.7	10.6
93	5	2	6.9	4.2	10.4	8.2	7.7	8.4	4.7	7.3	9.5	10.0	8.7	8.0
82	---	18	5.5	2.5	7.4	10.2	2.4	5.6	7.9	10.9	7.3	10.0	8.6	8.9

* Indicates that the sample broke during handling and could not be tested.

Table 3.7. Statistics from 25°C and 500°C shear tests in Table 3.6.

Braze Alloy (wt. %) Au Pd Ni			Average Failure Shear Stress (MPa)			
			25°C		500°C	
			Mean*	Std. Dev.*	Mean*	Std. Dev.*
---	60	40	17.1†	----	29.6	16.4
30	34	36	3.7	2.3	35.7†	----
50	25	25	----	----	----	----
70	8	22	107.3	53.9	114.7	11.5
93	5	2	78.9	24.5	87.4	23.1
82	---	18	60.4	36.3	97.2	16.0

* Samples which broke during handling were not included in these figures.

† No standard deviation is reported, only one sample was successfully tested.

Table 3.8. Failure mode of shear coupons. C & I indicates that the crack propagated in the ceramic parallel to the interface, I indicates that the crack propagated along the braze/ceramic interface.

Braze Alloy (wt%)			Failure Mode	
Au	Pd	Ni	25°C	500°C
---	60	40	I, C & I	I, 30% unbonded area
30	34	36	C & I	C & I
50	25	25	C & I	C & I
70	8	22	C & I	C & I
93	5	2	C & I	???, samples not completely broken
82	---	18	C & I	I, C & I

3.2.2.1.3 Reactions between Ti and Zr-coated Si₃N₄ with Pd-Containing Brazes

The results of the wetting tests on Ti-coated SWN1000 Si₃N₄ have been reported in the Phase I final report. All of the Pd-containing braze alloys studied wet the Ti-coated Si₃N₄ well, reaching a contact angle of approximately 10° after 2 minutes. In general, the reactions between the braze alloys and Ti-coated Si₃N₄ were moderate. The Ti-coating was observed to react with Si₃N₄ to form TiN [12,13]. Wetting tests were also performed on Zr-coated Si₃N₄ substrates. Severe gas evolution was seen for the Pd-containing brazes on Zr-coated Si₃N₄, and the wetting tests were not completed.

A series of experiments designed to study the interaction between braze alloys and the coated Si₃N₄ were run in a furnace/mass spectrometer unit. More N₂ gas was evolved when the brazes were melted on Zr-coated Si₃N₄ as compared to Ti-coated Si₃N₄. The difference in behavior between Ti- and Zr-coated Si₃N₄ was unexpected since Ti and Zr are both Group IVB elements and have many similar chemical characteristics. The gas evolution was not caused by a direct reaction between Pd and Si₃N₄ since Pd does not reduce Si₃N₄ to form a palladium silicide and N₂. The large amount of gas evolved during the wetting tests could not be explained by the comparatively small amount of residual gas initially present in the braze foils.

There was an important difference in the behavior of the Ti-coating

and Zr-coating on Si_3N_4 . Ti reacted with the Si_3N_4 to form a $2.5\text{ }\mu\text{m}$ thick titanium nitride layer, possibly composed of two forms of titanium nitride. However, the reaction between Zr and Si_3N_4 was a dissolution of N into the Zr without compound formation [8]. The Zr-rich region was $1.8\text{ }\mu\text{m}$ wide in this joint.

The difference in outgassing behavior between the Ti- and Zr-coated Si_3N_4 can be explained by the microstructures described above. When Ti reacted with Si_3N_4 , one or two layers of a titanium nitride compound were formed, and the N was tied up by the Ti. The titanium nitride layer acted as a diffusion barrier and slowed further reaction between the Ti and braze with the Si_3N_4 . However, when Zr reacted with Si_3N_4 , no zirconium nitride layer was formed.

3.2.2.2 Ni-based alloys

As mentioned previously, solid solution strengthened Ni-Cr-Fe and Ni-Cr-Mo alloys were chosen as master alloys for the high-temperature brazing alloys because of their excellent high-temperature strength and ductility. Some of the important requirements for the design of high-temperature brazing alloys are (1) brazability, (2) good ductility and low yield strength, (3) high-temperature creep strength, and (4) melting point in the vicinity of 1200°C . Design strategies are to find alloy compositions which satisfy the requirements above and to regain the mechanical properties of the master alloys as much as possible.

Two classes of alloying elements were considered in order to lower the melting points of the master alloys with minimum loss of mechanical properties; brittle metalloids such as B, Si, and Ge in the range of 1%-10% and ductile elements such as Au, Ag, Cu, and Pd in the range of 1%-30%. Within this compositional range, 100 new high-temperature brazing alloys were produced and evaluated with Differential Thermal Analysis (DTA), Rockwell hardness, and tensile testing.

Table 3.9 shows that the melting points and hardness of various Ni-based braze alloys with 1%-7% B, Si, and Ge. Differential Thermal Analysis (DTA) revealed that the additions of 3%-5% B or 6%-9% Si to the Ni-Cr-Fe alloy resulted in the melting points of $1000\text{-}1070^\circ\text{C}$ and $1110\text{-}1140^\circ\text{C}$, respectively, while the additions of the same quantities of B or Si to the Ni-Cr-Mo reduced the melting points to $960\text{-}1060^\circ\text{C}$ and $1130\text{-}1170^\circ\text{C}$, respectively. Both elements

were found to be effective in reducing the melting points of the alloys and maintaining the liquidus/solidus gap to less than 50°C. However, when the Rockwell hardness tests were performed, in general, the additions of B or Si increased the hardness values by 10%-50% compared to the master alloys, making the systems hard and brittle. It is well documented that the metalloid, like B, segregates on the grain boundary due to very low solubility in Ni, resulting in low melting points and high hardness. It is likely to see the formation of the compound, Ni_3B , in Ni+1%-5%B according to the phase diagram. When borides precipitate on the grain boundaries, the corrosion resistance of the alloys deteriorates significantly. The severe corrosion attack is expected due to the galvanic difference between the Ni matrix and the borides. Similar effects were reported for Si even though it has a higher solid solubility (7-8 w/o) in Ni. Germanium was also added to the master alloys to reduce the melting points. However, the level of reduction in the melting points was not significant compared to the results of B or Si, and no further investigation was pursued.

Table 3.9. Melting points and Rockwell hardness of various Ni-based braze alloys with 1%-7% of B, Si, and Ge.

Sample No.	Compositions* (wt%)	Solidus/Liquidus (°C)	Hardness† (R _B)
1	(NiCrFe) cast	1354/1413	67.2
2	(NiCrFe)-1B	1259/1316	99.1
3	(NiCrFe)-3B	1002/1043	96.3
4	(NiCrFe)-5B	1008/1073	117.4
5	(NiCrFe)-1Si	1371/1414	70.8
6	(NiCrFe)-5Si	1311/1374	-
7	(NiCrFe)-6Si	1205/1308	106.8
8	(NiCrFe)-9Si	1112/1138	118.1
9	(NiCrFe)-5Ge	1280/1306	-
10	(NiCrFe)-7Ge	1296/1316	-
11	(NiCrMo) cast	1290/1350	91.1
12	(NiCrMo)-1B	1154/1269	106
13	(NiCrMo)-3B	1042/1177	110.4
14	(NiCrMo)-5B	957/1064	119.7
15	(NiCrMo)-1Si	1295/1323	94.6
16	(NiCrMo)-3Si	1310/1381	71.9
17	(NiCrMo)-5Si	1136/1180	106.4
18	(NiCrMo)-7Si	1131/1171	100.9
19	(NiCrMo)-5Ge	1081/1279	-

*(NiCrFe) and (NiCrMo) are the master alloys representing (Ni-16Cr-7Fe) and (Ni-22Cr-9Mo-4Fe-3Nb), respectively. These are balanced in weight percent with additive elements.

†Values are average of five measurements.

In contrast, additions of IB alloying elements like Au or Cu (<10%) did not change the hardness values of the alloys noticeably, Table 3.10. The changes in hardness due to these elements were in the range of -10% to 10% of the master alloys. However, the addition of Ag to the master alloys made the alloys brittle. The different behavior between Au (or Cu) and Ag in hardness was attributed to the solubilities of these elements in Ni; Au or Cu forms a solid solution over the entire composition range, while Ag has a limited solubility in Ni (3 w/o at 1435°C).

The additions of 5% Cu, Au or Pd to the Ni-Cr-Fe master alloy reduced the

melting points by 50-70°C whereas 5% Ag reduced it by 30-50%. For the Ni-Cr-Mo alloy, the same additions reduced the melting temperature 70-90°C. Sample #16 in the Table 3.9 seems to be erratic due to inhomogeneous mixing of the alloy by arc melting. In general, the addition of 1%-7% of Au, Cu, Pd, or Ag did not satisfy the requirement for the melting point.

Preliminary tensile testing was done with some of Ni-Cr-Mo-Au alloys. The alloys were prepared using by arc melting, remelting 4 times to homogenize the alloys. Small-size specimens which are proportional to a standard size with a circular cross section of 0.16" in diameter were made according to ASTM Standard E8. Results of tensile testing at room-temperature are shown in Table 3.10.

Table 3.10. Melting points and Rockwell hardness of various Ni-based braze alloys with 1%-7% of Au, Cu, Pd, and Ag.

Sample No.	Compositions* (w/o)	Solidus/Liquidus (°C)	Hardness (R _B)	Tensile Strength (ksi)	Yield Strength† (ksi)	Elong. (%)
1	(NiCrFe) cast	1354/1413	67.2	75.2	23.8	74.7
2	(NiCrFe)-1Cu	1282/1336	68.1			
3	(NiCrFe)-5Cu	1309/1343	102.1			
4	(NiCrFe)-7Cu	1313/1349	61.7			
5	(NiCrFe)-5Au	1285/1338	77.5			
6	(NiCrFe)-5Pd	1305/1337	-			
7	(NiCrFe)-1Ag	1305/1353	-			
8	(NiCrFe)-5Ag	1324/1360	-			
9	(NiCrFe)-7Ag	1346/1389	-			
10	(NiCrMo) cast	1290/1350	91.1	101.8	29.7	77.3
11	(NiCrMo)-1Cu	1225/1293	101			
12	(NiCrMo)-5Cu	1221/1278	83.8			
13	(NiCrMo)-7Cu	1200/1281	-	96.8	20.7	54.8
14	(NiCrMo)-5Au	1210/1251	107.9	105.4	19.6	53.5
15	(NiCrMo)-5Pd	1231/1273	-			
16	(NiCrMo)-5Ag	843/875	-			

*(NiCrFe) and (NiCrMo) are the master alloys representing (Ni-16Cr-7Fe) and (Ni-22Cr-9Mo-4Fe-3Nb), respectively. These are balanced in weight percent with additive elements.

†Values are average of 2-4 testings.

The effects of Au and Cu on the tensile properties of the master alloy were similar, however addition of Au resulted in a higher ultimate tensile strength (UTS) than that of the Ni-Cr-Mo-Cu alloy. Compared to the arc melted master alloy, the change in UTS was almost negligible, while significant reductions in yield strength (about 60%) were noted for both alloys. The elongation values were maintained high at 50% even though they were lower than that of the Ni-Cr-Mo master alloy (70%). The tensile results are averages of 2-4 tests. Low yield strength alloys with high ultimate tensile strength are desirable for high-temperature brazing alloys since the braze alloys can accommodate the deformation associated with thermal mismatch between ceramic and metal.

Additional new braze alloys with higher percentages of Au, Cu, and Pd (10-20%) were produced to find optimum compositions in terms of melting points and tensile properties. DTA and mechanical testing results are shown in Tables 3.11 and 3.12. These results showed that the addition of 10%-20% IB elements or Ge to the Ni-Cr-Mo system resulted in melting points of 1220-1310°C and 970-1260°C, respectively, while the same additions of IB or Ge to the Ni-Cr-Fe system reduced the melting points to 1280-1380°C and 980-1320°C, respectively. The effects of these alloying elements on the melting points of the Ni-Cr-Fe alloys were found to be about the same as on the Ni-Cr-Mo alloys. Germanium reduced the melting points of the alloys significantly, even though the effect is not comparable with that of B or Si. Overall results indicated that the addition of Au, Cu, or Pd up to 20% still did not provide an adequate range of melting temperatures. The addition of Ge caused a broader melting range and multiple exothermic reactions near the melting points, implying the formation of various phases. This may limit the use of Ge as a component.

All additive elements used in this study increased the strength and the hardness of the alloys at the expense of elongation values when those were added to Ni-Cr-Fe or Mo. The hardness values in parentheses represented the elongation measured directly from the sample surfaces. It was noted that the Ge addition made the alloy brittle. Au seemed to be the most effective additive in maintaining excellent ductility without a loss of mechanical strength. Deformability of an alloy, which can be expressed quantitatively in a strain hardening factor, bound by ultimate tensile and yield strength values determines the survival of ceramic-metal joints at room- and high-

temperature. The ideal braze alloy should have a low strain hardening factor along with a large difference between ultimate tensile and yield strengths.

On the other hand, the tensile properties of the Ni-Cr-Mo alloy summarized in Table 3.12 showed excellent properties in general. Again, the Au-containing alloy exhibited the best properties. Combination of high ultimate tensile strength and extremely large elongation of this alloy will provide the ability to accommodate the stress built between ceramic and metal during processing and service. Pd-containing alloys behaved similarly to the Au-containing alloys. The Cu-containing alloys also resulted in reasonably good tensile properties, even if the properties degrade as the Cu content increases. The Ni-Cr-Mo alloys showed a higher strength level than that of Ni-Cr-Fe alloys. The addition of Au to the system resulted in the best properties among the alloys.

Table 3.11. Physical properties of Ni-Cr-Fe-10%-20% IB alloys.

Sample No.	Compositions* (w/o)	Solidus/Liquidus (°C)	Hardness (RB)	Tensile Strength* (ksi)	Yield Strength† (ksi)	Elong. (%)
1	(NiCrFe)-10Au	1311/1369	85.4	97.1	-	99(63) *
2	(NiCrFe)-15Au	1317/1348	91			
3	(NiCrFe)-20Au	1283/1330	93.4			
4	(NiCrFe)-10Ge	1300/1324	86.3	98.6	15.7	79(31)
5	(NiCrFe)-15Ge	1008/1262	101.7			
6	(NiCrFe)-20Ge	984/1007	113			
7	(NiCrFe)-10Cu	1343/1375	70.1	75	11.5	97(46)
8	(NiCrFe)-15Cu	1297/1384	68.5	76.5	12.8	92(54)
9	(NiCrFe)-20Cu	1297/1336	75.8			
10	(NiCrFe)-10Pd	1298/1380	84.6			
11	(NiCrFe)-15Pd	1293/1340	89.3			
12	(NiCrFe)-20Pd	1279/1327	88.1			

* (NiCrFe) and (NiCrMo) are the master alloys representing (Ni-16Cr-7Fe) and (Ni-22Cr-9Mo-4Fe-3Nb), respectively. These are balanced in weight percent with additive elements.

† Measured elongations from tested samples

Table 3.12. Physical properties of Ni-Cr-Mo-10-20% IB alloys

Sample No.	Compositions* (w/o)	Solidus/Liquidus (°C)	Hardness (RB)	Tensile Strength* (ksi)	Yield Strength† (ksi)	Elong. (%)
1	(NiCrMo)-10Au	1304/1312	94.1	106.2	15.6	104
2	(NiCrMo)-15Au	1266/1276	95.2			
3	(NiCrMo)-20Au	-	95.4			
4	(NiCrMo)-10Ge	1109/1266	96	75.7	18	30(5)*
5	(NiCrMo)-15Ge	1097/1169	106.2			
6	(NiCrMo)-20Ge	974/991	115.2			
7	(NiCrMo)-10Cu	1262/1299	85.2	94.9	13.3	105
8	(NiCrMo)-15Cu	1205/1286	88	65.8	16.1	43
9	(NiCrMo)-20Cu	1220/1271	87.6	63	13.5	70
10	(NiCrMo)-10Pd	1274/1310	90.2	105.7	15.4	102
11	(NiCrMo)-15Pd	1222/1299	90.9			
12	(NiCrMo)-20Pd	1235/1301	91.6			

*(NiCrFe) and (NiCrMo) are the master alloys representing (Ni-16Cr-7Fe) and (Ni-22Cr-9Mo-4Fe-3Nb), respectively. These are balanced in weight percent with additive elements.

*Measured elongations from tested samples

Based on the results above, further efforts were made to reduce the melting points of the alloys. Other studies described in this report also indicated that processing temperature for ceramic-metal joints was a more stringent requirement than others such as strength, ductility, and use-temperature in developing new braze alloys. Incoloy 909 loses its desirable mechanical properties if heat treated above 1200°C. Also, our recent coating study revealed that severe degassing began around 1200°C from the Ti- or Zr-coated PY6 silicon nitride. The degassing can cause gas entrapment in the joining area, resulting in a large unbonded area. Therefore, it is recommended that all brazing processes be carried out below or at 1200°C.

Also, finite element analysis (FEA), summarized in the proceeding section of this report, showed that brittle braze alloys such as alloys of intermetallic compositions increase the maximum tensile stress in the vicinity of the joint 5-7 times compared to a ductile braze alloy like Au-Pd-Ni. The FEA

study with various braze alloys and interlayer materials indicated that alloys with a high thermal expansion coefficient and low yield strength would perform better in handling the residual stress at the ceramic/metal interface than those with a low thermal expansion coefficient, high yield strength, and high Young's modulus. These results emphasize the significance of high ductility of the brazing alloys and interlayer materials.

Based on this information, another set of new brazing alloys were designed to attain a liquidus temperature around 1150°C with a low yield strength. Also, effort was made for new alloys to increase the Ni content as much as possible to improve high-temperature performance. As base compositions, 10% of Au, Cu, Pd, or Ge was added to Ni-Cr-Fe and Ni-Cr-Mo. Then, metalloids in the range of 1%-7% B, Si, and Ge were added to these compositions. Indium and Sn have been frequently used in the Ag- or Au-based brazing alloys to lower the melting points with a moderate loss of ductility. Therefore, 5-15% In or Sn were alloyed to Ni-Cr-Fe-Mo-Cu or Au.

The addition of B, Si, or Ge to Ni-Cr-Fe-Mo-Cu or Au brought the melting temperatures close to the target range, i.e., 1100-1200°C. However, their addition caused a significant loss of ductility, except for the Ge addition. Further hardening of the alloys was seen when B and Si were added together to the base composition. A similar effect was observed from Ni-Cr-Fe-10% Cu, Pd, or Ge.

The results of DTA study in Table 3.13 showed that the addition of 10% Sn or 5-10% In to Ni-Cr-Mo-Cu alloys has been effective in lowering the melting points below 1200°C. The additions of 10% Sn or 5% In resulted in melting points of 915-1180°C and 1040-1210°C, respectively. The large temperature gap between solidus and liquidus ($\Delta T_{eq} > 150^\circ\text{C}$) was a setback of these alloys. Further, Rockwell hardness tests showed R_B values over 100, while useful ductility for Ni-Cr-Mo alloys was found around 90. Also, the hardness decreases as the Cu content increases. No further investigations were pursued because of the embrittling effect of Sn and In in the alloys.

Table 3.13. Properties of Ni-Cr-Mo-IB-B, Si, Ge, Sn, and In alloys.

Sample No.	Compositions* (w/o)	Solidus/Liquidus (°C)	Hardness (RB)	Tensile Strength† (ksi)	Elong. (%)
1	(NiCrMo)-10Au	1282/1310	94		
2	(NiCrMo)-10Au-1B	1104/1200		122.2	29(30)
3	(NiCrMo)-10Au-3Si	1044/1240			
4	(NiCrMo)-10Au-3Ge	1184/1280			
5	(NiCrMo)-10Cu	1262/1299	85		
6	(NiCrMo)-10Cu-1B	1140/1219		121.9	33(11)
7	(NiCrMo)-10Cu-3Si	1123/1283		99.2	33(8)
8	(NiCrMo)-10Cu-3Ge	1191/1294		101	91(60)
9	(NiCrMo)-10Au-1B-3Si	985/1086		>50	F.G.*
10	(NiCrMo)-10Cu-1B-3Si	1000/1154		97	24(3)
11	(NiCrMo)-10Pd-1B-3Si	1021/1181		>84	F.G.
12	(NiCrMo)-10Ge-10Ni	1084/1301		80.7	37(14)
13					
14	(NiCrFe)-10Cu-2B-5Si	921/1100		>84	
15	(NiCrFe)-10Pd-2B-5Si	815/1102			hard
16	(NiCrFe)-10Ge-2B-5Si	977/1041			hard
17	(NiCrMo)-10Au-1B-2Si	1043/1171	105		
18	(NiCrMo)-10Au-1.5B-3Si	986/1109	109		
19	(NiCrMo)-10Cu-1B-2Si	1042/1095	104		
20	(NiCrMo)-10Cu-1.5B-3Si	941/971	110		
21	(NiCrMo)-10Cu-5Sn	1156/1267	98		
22	(NiCrMo)-10Cu-7Sn	1140/1259	97		
23	(NiCrMo)-10Cu-5Sn-3Ge	1113/1236	97		
24	(NiCrMo)-10Cu-7Sn-3Ge	950/1186	101		
25					
26	(NiCrMo)-10Cu-10Sn	960/1182	102		
27	(NiCrMo)-10Cu-15Sn	893/943	107		
28	(NiCrMo)-10Cu-10Sn-3Ge	878/920	101		
29	(NiCrMo)-10Cu-15Sn-3Ge	880/931	109		
30	(NiCrMo)-10Cu-5In				
31	(NiCrMo)-10Cu-10In		102		
32	(NiCrMo)-10Cu-5In-3Ge	1086/1209	96		
33	(NiCrMo)-10Cu-10In-3Ge	1042/1190	98		
34	(NiCrMo)-20Cu-3In		87.7		
35	(NiCrMo)-20Cu-5In		94.5		
36	(NiCrMo)-20Cu-3In-3Ge		92.3		
37	(NiCrMo)-20Cu-5In-3Ge		80.6		
38	(NiCrMo)-30Cu-3In		88.6		
39	(NiCrMo)-30Cu-5In		90.5		
40	(NiCrMo)-30Cu-3In-3Ge		92.4		
41	(NiCrMo)-30Cu-5In-3Ge		88.8		

* (NiCrFe) and (NiCrMo) are the master alloys representing (Ni-16Cr-7Fe) and (Ni-22Cr-9Mo-4Fe-3Nb), respectively. These are balanced in weight percent with additive elements.

† Sample failed at the grip area.

3.2.2.3 Au-based alloys

Tailoring of Ni-based alloys to meet the required brazing temperature by introducing metalloids into the system has shown unacceptable losses of ductility. Meanwhile, additions of IB elements up to 20% were not enough to reduce the brazing temperature below 1150°C. It was intended with Au-based alloys that new brazing alloys would be designed by adding high-temperature strengtheners such as Ni, Cr, Fe, Mo, Nb, and V to the system. Gold-nickel alloys were chosen as base compositions, expecting that two phase structure due to the extensive miscibility gap in Au-Ni system might provide high-temperature creep strength if other high-temperature strengtheners tend to affiliate with Ni or Au. Selected ratios of Au to Ni were in the range of 1.0-6.5 to provide appropriate melting points.

Thus, Au-Ni-Cr-Mo and Au-Ni-Cu-Cr-Mo alloys were produced to meet the processing temperature and yield strength requirements with a moderate gain in high-temperature creep strength. These alloys provided desirable melting temperatures (<1200°C) and ductility for ceramic-metal joints. They are expected to show improved high-temperature performance compared to previous alloys like Au-Pd-Ni and Au-Ni due to the presence of 20%-40% Ni-based superalloy in the system. Optimization of these alloys will be done by increasing Ni content and replacing Cr content with other solid solution strengtheners like Mo and Fe. Oxidation resistance which is obtained from Cr for Ni-based alloys will be maintained by the presence of Au in the system.

As shown in Tables 3.14 and 3.15, various Au-Ni-Cr alloys were produced by induction melting in argon atmosphere and characterized in terms of wetting and ductility. Wetting of these new braze compositions on the Ti-coated ceramic substrate was found to be excellent in general, however, wetting behavior of the braze alloys on Ni or Inconel 600 substrate varied significantly depending on the melting points and braze compositions. Brazed coupons were made of PY6 and Inconel 600 to see the compatibility of the braze system during and after the brazing process using new alloys.

Au-Ni-Cr-Fe-Mo-Nb alloys (A-C alloys) were produced maintaining the Mo to Fe ratio of 2.2. All compositions exhibited excellent ductility while good flow (wetting on Inconel 600) was observed only from C alloys with low melting points (950-1050°C). For the compositions with low Au/Ni ratios, like A alloys, low amounts of Cr, Mo, and Fe are needed for low melting points. More than 10% of Cr, Mo, and Fe should be avoided unless the Au/Ni ratio exceeds 2.0.

The A1 composition was repeated in K1 alloy without Nb. Niobium was removed from the composition since the presence of Nb in the Ni-based alloys is known to cause hot cracking during solidification by forming Laves phases. No significant changes in the flow properties were noted from K1 alloy. Figure 3.5 shows a typical microstructure of cast K1 alloy; a discrete phase separation was observed. These phases consisted of Ni-Cr-Fe-Mo and Au-Ni-Cr-Fe which might enhance creep resistance without sacrificing ductility. It was noted from energy dispersive X-ray analysis (EDXA) that Cr, Fe, and Mo showed a high affinity for Ni, resulting in the Ni-rich phase accounting for more than 80% of the structure. The amount of Au in this alloy (40 wt%) corresponds to about 20 vol%. This implies that Au-Ni phase forms a solid solution with Fe and Cr within solid solubility limits. Since the microstructure of the systems mentioned above showed good potential to meet the requirements for the high-temperature application of ceramic-metal joints, further investigation was made with these alloy systems. However, the increase in Au content shown in L alloys did not significantly improve the flow property of K1 alloy.

Of those alloys, Au-Ni-Cr-Mo-Fe-Nb alloys which melt at 1050-1100°C (B alloys) seemed to meet the processing requirements for high-temperature ceramic-metal joints. Preliminary results from mechanical testing indicated that these alloys had good ductilities and strengths. They wetted well on silicon nitride with various coating materials in the vicinity of 1150°C. However, a major drawback was the limited wetting property of these alloys on Inconel 600 or Ni substrates due to the combined effects of liquation and alloying.

Therefore, the relative effects of Cr and Fe on ductility and the flow property of this system were evaluated by adding Cr, Fe and Mo systematically to the Au-18Ni system. It showed that (1) the addition of Fe to a Au-Ni system did not interfere with the excellent flow property of a Au-Ni system, but it reduced the ductility with an increase of Fe content, (2) high content of Cr in a Au-Ni system embrittled the braze alloy and reduced the flow characteristics of liquid braze on Inconel 600, (3) excessive addition of Mo to the Au-Ni system lowered the flow property significantly, and (4) the presence of Mo and the ratio of Au to Ni appeared to play important roles in enhancing the alloy ductility. The Fe embrittlement in this system was presumed to be due to the phase segregation or precipitation at low temperature. The reduction of flow

by the addition of Cr can be explained with an Au-Cr phase diagram as well; the melting point increases with the increase of Cr content. The role of Mo on the flow property was closely related to the limited solubility of Mo in Au. Further, the Mo addition caused substrate erosion resulting from dilution of base-metal. The solubility limits of Cr, Fe, and Nb in Au were reported to be 20.8, 46, and 38.5 wt%, respectively.

Some of the A-F0611 alloys which have no Mo in the compositions have showed improved flow along with ductility. B-D0611 alloys, which have the Au to Ni ratio of 1.5-2.1, showed the best results. These alloys contained less than 10 wt% of Cr, Mo, and Fe in total. Alloys A and E0617 proved again that high Cr content not only embrittled the alloys but reduced the wettability of the braze on the Ni substrate. Since the Mo addition improves the alloys' ductility, various Au:Ni and Mo:Fe ratios were tried to find the optimum compositions. Alloy E0618 Au:Ni ratio of which is 1.6 with the Mo:Fe ratio of 1.0 performed the best.

Based on the E0618 alloy, A-E0619 alloys were designed. These alloys maintained the same Mo:Fe ratio (1.0) as in Alloy E0618 with various amounts of Cr, Mo, or Fe. Alloys B, D, and E0619 were found to exhibit reasonable ductility and wetting. To optimize these compositions, alloys with various Mo:Fe ratios (0.3-1.0), the total contents of Cr, Fe, and Mo (5%-20%), and Au:Ni ratios (0.6-2.4) were investigated. It was determined from alloy screening that D0611, E0618, B, D, and E0619 were chosen as final candidates. Others were rejected due to the high melting points, low wettability, or low ductilities.

In addition, 22 Au-Ni-Cr-Cu-Mo-Fe-Nb and Au-Ni-Cu-Cr-Fe alloys were studied by adding Cu to Au-Ni-Cr-Mo-Fe-Nb and Au-Ni-Cr-Fe systems to improve the wetting property. Copper was chosen due to its complete solid solubility with Au. However, addition of Cu did not enhance the wettability of these alloys and raised the melting points of the alloys unexpectedly ($>1150^{\circ}\text{C}$). These alloys were not able to be processed at temperature less than 1200°C .

Table 3.14: Development of Au-Ni-Cr-Fe(-Mo) Alloy for Brazing

ALLOY	%Au	%Ni	%Cr	%Mo	%Fe	%Nb	Au/Ni	Mo/Fe	Cr+Mo+Fe	Ductility	Flow	Melting(°C)
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
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57												

Table 3.14. Development of Au-Ni-Cr-Fe(-Mo) alloy for brazing (continued)

	ALLOY	%Au	%Ni	%Cr	%Mo	%Fe	%Nb	Au/Ni	Mo/Fe	Cr+Mo+Fe	Ductility	Flow	Melting(°C)
58	E	57.9	33.9	3.9	2	2.1		1.7	1.0	8	Y7(Y2)	Y1	1050
59													
60	L1+2Fe	49	31.4	11.1	4.5	4.0		1.6	1.1	19.6	Y	N	1150
61	L3+1.5Fe	48.3	39.4	6.7	2.8	2.7		1.2	1.0	12.2	Y7	N2	1150
62	L4+1.5Fe	55.2	25.6	6.7	2.8	2.7		2.4	1.0	12.2	Y	N2	1150
63	L5+1.5Fe	55.2	32.5	6.7	2.8	2.7		1.7	1.0	12.2	Y	N2	1150
64													
65	(L1+2Fe)+2Fe	48	30.8	10.9	4.4	5.9		1.6	0.7	21.2	Y3	N1	
66	(L3+1.5Fe)+2Fe	47.3	38.6	6.6	2.7	4.6		1.2	0.6	13.9	Y1	N2	
67	(L4+1.5Fe)+2Fe	60.9	25.1	6.6	2.7	4.6		2.4	0.6	13.9	Y3	N2	
68	(L5+1.5Fe)+2Fe	54.1	31.9	6.6	2.7	4.6		1.7	0.6	13.9	Y3	N2	
69													
70	A0628	55	35		5	5		1.6	1.0		N	N	
71	B	55	35		2.5	7.5		1.6	0.3		N	N	
72	C	55	35	2	4	4		1.6	1.0	10	Y2	N1	
73	D	54	32	3.5	3.5	7		1.7	0.5	14	Y3	N	
74	E	55	33	3	3	6		1.7	0.5	12	Y3	N	
75	F	56	34	2.5	2.5	5		1.6	0.5	10	Y2	N	
76	G	55	37	2	2	4		1.5	0.5	8	Y2	N1	
77	H	57	35	2	2	4		1.6	0.5	8	Y2	N	
78													
79	A0701	40	46.2	9		4.8		0.9		13.8	Y3	N	>1175
80	B	36	50.2	9		4.8		0.7		13.8	Y3	N	>1175
81	C	32	54.2	9		4.8		0.6		13.8	Y3	N	>1175
82	D	45	43.5	7.5		4		1.0		11.5	Y2	Y1	>1175
83	E	40	48.5	7.5		4		0.8		11.5	Y2	N	>1175
84	F	35	53.5	7.5		4		0.7		11.5	Y2	N	>1175
85	*G	48	42.8	6		3.2		1.1		9.2	Y3	Y1	<1100
86	H	42	48.8	6		3.2		0.9		9.2	Y3	N1	>1100
87	I	36	54.8	6		3.2		0.7		9.2	Y2	N1	>1100
88	J	42	51.1	4.5		2.4		0.8		6.9	Y2	N1	>1100
89													
90	*A0702	57.9	34.3	3.3	1.7	2.8		1.7	0.6	7.8	N	Y2	>1100
91	B	58.9	32.6	4.3	1.5	2.6		1.8	0.6	8.4	N	Y1	>1100
92	*C	57.3	34	4.7	1.6	2.4		1.7	0.7	8.7	N	Y2	>1100
93													
94	A0618R	56	35	6	1	2		1.6	0.5	9	N	N1	<1175
95	B	56	33	8	1	2		1.7	0.5	11	Y2	N1	-
96	*C	54	37	6	1	2		1.5	0.5	9	N	Y2	-
97	D	54	35	8	1	2		1.5	0.5	11	N	N2	-
98	E	56	38	4	1	1		1.5	1.0	6	N	N3	-
99	F	54	40	4	1	1		1.4	1.0	6	N	Y1	-
100													
101	*A0619R	55	38	5	1	1		1.4	1.0	7	Y2	Y3	-
102	B	55	37	6	1	1		1.5	1.0	8	Y2	N3	-
103	C	54	36	8	1	1		1.5	1.0	10	N	N3	-
104	D	56	37	4	1.5	1.5		1.5	1.0	7	Y2	N1	-
105	E	55	37	4	2	2		1.5	1.0	8	Y1	N1	-

Note: Y stands for yes and N stands for no. Y3 means the best performance

while N3 is the worst one.

Table 3.15: Development of Au-Ni-Cr-Cu-Fe Alloys for Brazing

	ALLOY	%Au	%Ni	%Cr	%Cu	%Mo	%Fe	%Nb	Column 9	Column 10	Ductility	Flow	Melting(°C)
1	A0610	45	31	11	5	4.5	2	1.5			Y	N	>1150
2	B	54	25	9	6	3.6	1.8	1.2			Y	N	>1150
3	C	63	19	7	7	2.7	1.2	0.9			Y	N	>1150
4	D	40	31	11	10	4.5	2	1.5			Y	N	>1150
5	E	48	25	9	12	3.6	1.8	1.2			Y	N	>1150
6	F	56	19	7	14	2.7	1.2	0.9			Y	N	>1150
7	G	35	31	11	15	4.5	2	1.5			Y	N	>1150
8	H	42	25	9	18	3.6	1.6	1.2			Y	N	>1150
9	I	49	19	7	21	2.7	1.2	0.9			Y	N	>1150
10	J	30	31	11	20	4.5	2	1.5			Y	N	>1150
11	K	36	25	9	28	3.6	1.6	1.2			Y	N	>1150
12												N	>1150
13	A0613	45	38	7.5	5		4				Y	N	>1150
14	B	54	31	6	6		3.2				Y	N	>1150
15	C	63	23	4.5	7		2.4				Y	N	>1150
16	D	40	38	7.5	10		4				Y	N	>1150
17	E	48	31	6	12		3.2				Y	N	>1150
18	F	56	23	4.5	14		2.4				Y	N	>1150
19	G	35	38	7.5	15		4				Y	N	>1150
20	H	42	31	6	18		3.2				Y	N	>1150
21	I	49	23	4.5	21		2.4				Y	N	>1150
22	J	30	38	7.5	20		4				Y	N	>1150
23	K	36	31	6	26		3.2				Y	N	>1150

As-Cast

Cr

Au

Fe

Ni

Mo

Figure 3.5. Typical microstructure of cast K1 alloy. A discrete phase separation was observed. These phases consisted of Ni-Cr-Fe-Mo and Au-Ni-Cr-Fe.

3.2.3 Selection of Final Composition and Alloy Production

Brazing experiments were performed to check the brazability and system compatibility of Alloys D0611, E0618, B, D, and E0619. Limited wetting of Au-Ni-Cr-Mo-Fe braze alloys resulted in spotty bonding between Ti-coated PY6 and Inconel 600, even though these alloys wetted Ti-coated PY6 completely. On the other hand, the Au-Ni-Cr-Fe system generally provided better wetting and bonding between PY6 and Inconel 600. Based on the results obtained above, two alloy compositions, D0619 and D0611, were selected for final foil production. D0619 and D0611 were designated as Alloys SK-1 and SK-2, respectively in the remainder of this paper. These alloys have shown superior wetting and bonding characteristics as well as excellent ductility compared to other compositions studied. These alloys melted in the vicinity of 1000°C and could be brazed at 1050-1100°C which people in auto industries feel as an upper limit for ceramic-metal joining process. The alloys, even though they have relatively low melting temperatures, are expected to improve high-temperature creep strength partially by diffusion of Ni, Cr, Fe and Mo from the adjacent interlayer (Ni or Inconel 600).

Braze foil production of two alloy compositions, Au-Ni-Cr-Mo-Fe and Au-Ni-Cr-Fe alloys, was completed at GTE Wesgo. One hundred twenty grams of each alloy was melted at 1300-1400°C in a boron nitride crucible by an induction heating and cast into a copper mold to produce 2.0" x 0.75" x 0.25" bar. The alloy was homogenized at 800°C for 24 hours in an argon atmosphere. The cast bar was rolled into 0.002" thick foil with intermittent vacuum annealing at 800°C for 2-4 hours. The rolling of this alloy was done successful due to its excellent toughness and ductility. The second batches of Alloys SK-1 and SK-2 were produced in the same way as done for the first batches of the alloys.

3.2.3.1 Chemical and Differential Thermal Analysis

Brazing foils produced in two separate batches were subjected to chemical analyses to identify the compositions of the alloys. The alloy foils were cleaned with acetone and dissolved in a solution mixture of concentrated sulfuric, nitric and hydrochloric acids. The compositions of the solutions were then analyzed using an ICP (inductively coupled plasma) analyzer. Each solution was examined more than three times and the values were averaged after background correction. Normalized alloy compositions are listed in Table

3.16. This result shows that there is insignificant difference in compositions between the first and second batch alloys. Also, the target compositions were obtained without noticeable deviation and alloy compositions were found to be uniform throughout the foils.

Differential thermal analysis (DTA) was done for the final compositions, SK-1 and SK-2 with a DuPont TA-2100 Analyzer. Figure 3.6 shows two DTA results obtained from heating and cooling cycles. Alloy SK-1 exhibited two discrete melting activities at 1005°C and 1260°C. Alloy SK-2 also showed exactly the same behavior as SK-1 at 1005°C and 1270°C even though the melting phenomenon at high-temperature was not as clear as at low temperature. Two melting points of an alloy implied the existence of two different phases in the microstructure. This was proved in the following section, showing Au-based and Ni-based phases. The Au-based phase melted in the vicinity of 1000°C and the Ni-based phase at the temperature range of 1260-1270°C.

Table 3.16. Compositional analysis of high-temperature braze alloys.

Alloys	Au	Ni	Cr (wt%)	Fe	Mo	Batch
SK-1	58.5	34.6	3.9	1.40	1.50	target
	56.9	35.8	4.03	1.57	1.70	1
	60.8	32.4	3.84	1.49	1.50	1
	59.5	33.6	3.85	1.54	1.56	2
	59.7	33.3	3.93	1.52	1.57	2
	59.7	33.1	4.08	1.51	1.55	2
SK-2	56.0	36.0	4.50	2.40	-	target
	57.2	35.7	4.87	2.23	-	1
	57.3	35.8	4.73	2.18	-	1
	57.4	35.5	4.95	2.16	-	2
	58.0	34.7	5.05	2.19	-	2
	57.2	35.5	5.03	2.18	-	2

Figure 3.6. DTA results obtained from heating and cooling cycles of the (top) SK-1 alloy and (bottom) SK-2 alloy.

3.2.3.2 Microstructural Analysis

The microstructure of PY6/Ni/Incoloy 909 joints made with SK-1 and SK-2 alloys was analyzed. A JEOL 840W microprobe was used to characterize the microstructures. The general microstructure of the joint made with SK-1 showed two discrete Au- or Ni-based phases, as shown in Figure 3.7. This microstructure differed from that of the K1 alloy, Figure 3.5, even though both alloys contained two phases. The K1 alloy showed a continuous Au-rich phase with a rounded Ni-rich phase floating inside the matrix while the SK-1 alloy exhibited a continuous Ni-rich phase with a rugged Au-rich phase floating inside. The reason the SK-1 alloy has more than 55% Au and shows the segregation of the Au-rich phase from the Ni-rich phase is not known.

The microstructures of brazed joints consistently showed a fine layer of the Au-rich phase at the braze/ceramic interface. This reveals that the bonding between ceramic and Ni interlayer relies on the spreading of the phase with the lowest melting temperature, the Au-based phase. This phase was not found at the braze/interlayer interface. Creep might preferential occur in the low melting temperature Ar-rich phase. Especially, the thin layer of Au-rich phase at the interface could start deforming at a much lower stress level due to the localized strain effect. The discontinuity of the Au-rich phase in the structure or at the interface might increase high-temperature creep resistance by involving Ni-rich phase in the deformation.

EDXA (energy dispersive X-ray analysis) results show that the Au-rich phase in SK-1 consists of Au, Ni, Fe, and Mo. Since Mo showed a nonuniform distribution inside or in the vicinity of the Au-rich phase, Mo seemed to remain as pure Mo in the system. A low solid solubility limit of Mo in Au, almost 0% at RT and 1.0% at 1054°C, supports this explanation. Most of the Ti-coating layer remained at the interface. The Ni-rich phase consists of Ni-Cr-Fe in the form of a solid solution. This phase is needed for high strength and high ductility at high-temperatures. A small amount of Mo was also present in this Ni-Cr-Fe phase.

A similar observation was made from the SK-2 alloy (Figure 3.8). However, this alloy contained a much higher volume fraction of the Au-rich phase than did the SK-1 alloy, which has 58.5% Au. Also, the Au-rich phase was found to be more or less continuous, as in the K1 alloy. EDXA results showed similar elements in solution in the Au-rich and Ni-rich phases. One of the possible explanations for the increased volume fraction of this Au-rich

phase is that the Au-rich phase in this alloy system has a stronger affinity with Ni than in the SK-1 alloy. The high volume fraction of Au phase in SK-2 was responsible for the excellent wetting behavior of SK-1 at the interface. The Ti-coating remained intact to the PY6 ceramic without totally dissolving in the Au-rich phase. The Ni-rich phase was a solid solution of Ni-Cr-Fe.

Analytical electron microscopy (AEM) was done for the SK-1 foil which was heated to 1100°C for 30 minutes to resemble the brazing process; the results are shown in Figure 3.9. This figure shows clearly that the alloy consisted of two solid solution phases, a Ni-Cr-Fe-Au phase of light contrast and a Au-Cr-Fe-Ni phase of dark contrast, both were without precipitates. A type of lamellae structure is seen. As a result of cold rolling during foil production, a high density of dislocations were found in the Ni-rich phase. Also, microdiffraction analysis confirmed that the Au-rich and Ni-rich phases have the same preferred orientation, probably from the formation of texture along the rolling direction. Figure 3.10 shows the microstructure of SK-2 which was melted and held at 1100°C for 30 minutes to resemble the microstructure of a braze joint. Fewer dislocations were noted without change in morphology.

Secondary

Mo

Au

Fe

Ni

Cr

Figure 3.7. SEM image and X-ray maps of SK-1 braze.

Secondary

Cr

Au

Fe

Ni

Ti

Figure 3.8. SEM image and X-maps of SK-2 brake.

Figure 3.9. AEM image of SK-1 braze: Strip-like phase is the Au-rich phase.

Figure 3.10. AEM image of SK-2 braze: Thin layer is the Au-rich phase.

3.3 Coating Development

3.3.1 Effect of Double Coating Layers

For phase I of the program, Ti was used as a coating material for PY6 silicon nitride. The thickness of the coatings was about 3.0 μm . In the previous study, the adhesion strength of the Ti-coating was found to degrade significantly as the brazing temperature increased to 1200°C. Therefore, the effect of coating thickness on the joint performance was investigated with double-layer coatings, varying the thickness of the Ti-coating. The double-layer coatings composed of layers of a reactive metal and a nonreactive metal were intended to keep the Ti-coating from extensive oxidation during the brazing process. A Ag-coating of 0.2 μm in thickness was deposited on the top of the Ti-coating by electron beam evaporation. The thickness of Ti was in the range of 0.5-6.0 μm . High-temperature performance tests were done at 1300°C for 2 hours with or without a braze alloy. Simple scratch test results showed that 0.5-1.5 μm thick Ti-coating provided better adhesion strength than the 5.75 μm thick coating.

Unreacted Ti in the 5.75 μm thick coating caused spalling of the coating layers. The experiments with braze alloys (Ni-Cr-Mo-Cu) showed that the wetting of high-temperature braze alloys improved as the coating thickness increased. However, the excessive amount of unreacted Ti tends to separate the braze alloy completely from the ceramic substrate. Based on the wetting and strength results, about 1.5-3.0 μm thick Ti was determined to be an optimum.

Additional tests were done to evaluate the materials for the second layer coatings. Silver, Cu, Ni, and Al were selected on the basis of their melting points and materials characteristics. The thicknesses used for the Ti-coatings and the second layer coatings were 1.5 μm and 0.2 μm , respectively. Wetting experiments were done with a Ni-Cr-Mo-Cu alloy. Little difference was found among these materials as second coating layers in terms of adhesion strength and wetting. All exhibited excellent wetting and adhesion strength after being heat-treated at 1300°C for 2 hours in vacuum. It was presumed that the thin layers of these coating materials dissolved into braze alloys without significantly influencing the adhesion performance.

3.3.2 Coating/Braze Alloy Interaction

Section 3.2.2.1.3 reports that the interactions between Ti-coating and Pd-containing alloys caused significant processing problems by degassing phenomena. A similar experiment was performed with alloys C2, B5, Cu, and Au in the temperature range of 1000-1250°C. Testing was done with 3 μm Ti-coating and Zr- or double coatings such as Ni-Zr, Ni-Hf, Ni-Ti, Au-Zr, Au-Ti, Cu-Zr, and Cu-Ti. One-half gram of each alloy was melted on the coated PY6 substrates in an Ar atmosphere, and degassing was monitored as a function of temperature, Table 3.17. Degassing was observed in all cases with Au-Ni-Cr alloys regardless of the coating materials. Significant reactions started in the vicinity of 1170°C. Liquid Au or Cu did not cause any signs of degassing in this temperature range. Since Ni is the only element which overlaps between alloys in section 3.2.2.1.3 and the Au-Ni-Cr in this study, Ni could be suspected as another catalyzing element to cause this reaction at this temperature range. However, it was not confirmed with additional experiments.

Table 3.17. Braze/coating interaction for coated PY6 silicon nitride.

Sample No.	Alloy	Coating (μm)	Temperature ($^{\circ}\text{C}$)	Degassing
1	C2	3 Ti	1000/1240	Y
2	"	3 Zr	1100/1210	Y
3	"	1.5 Ni/1.5 Zr	1080/1250	Y
4	B5	3 Ti	"	Y
5	"	3 Zr	"	Y
6	"	1.5 Ni/1.5 Zr	"	Y
7	C2	1.5 Ni/1.5 Hf	"	Y
8	"	0.2 Ni/1.5 Ti	1060/1240	Y
9	"	0.2 Ni/1.5 Zr	"	Y
10	"	0.2 Au/1.5 Ti	1075/1250	Y
11	"	0.2 Au/1.5 Zr	1040/1240	Y
12	"	0.2 Cu/1.5 Ti	1050/1250	Y
13	"	0.2 Cu/1.5 Zr	"	Y
14	Pure Cu	1.5 Ni/1.5 Zr	1090/1325	N
15	Pure Au	1.5 Ni/1.5 Zr	1030-1245	N

4. FINAL JOINT SYSTEM

4.1 Effect of Joint Geometry

Various wall thicknesses and interlayer thicknesses were examined to reduce the overall residual stress developed at the ceramic-metal interface. The range of the thickness values of interlayers was obtained from the results of FEA study in Phase I. According to the calculation, the stress level was at a minimum if the Ni interlayer thickness was more than 1 mm. Also, the wall thickness of Incoloy 909 was reduced to the minimum level where the applied torque was slightly lower than the yield stress of the Incoloy 909. Table 4.1 shows the maximum torque as a function of the wall thickness of Incoloy 909 at the onset of yielding. It was expected that the reduction of wall thickness would decrease the magnitude of the shrink fit effect (radial compressive stress). Table 4.2 shows the changes in torsional strength of the PY6/Ni/Incoloy 909 joints with dimensional changes in wall thickness and interlayer thickness. In general, the joints made with the reduced wall thicknesses resulted in significant misalignment, causing a dramatic reduction in joint strength. It was difficult to separate the effects of thin interlayers and thin walls due to the effect of misalignment. Based on this study, it was concluded that the dimensions used in Phase I of the program were suitable for joint production and strength. It is generally recommended by FEA studies to have thicker interlayers if the interlayer materials are of low yield strength and high ductility.

Table 4.1. Stress in Incoloy 909 as a function of wall thickness and torque.

Wall Thickness (in)	Torque (in-lbs)	Shear Stress (ksi)
0.010	180	27.4
	260	39.6
	340	51.8
0.020	180	13.5
	260	19.5
	340	25.5
0.030	180	8.8
	260	12.8
	340	16.7
0.040	180	6.5
	260	9.4
	340	12.3
0.050	180	5.1
	260	7.4
	340	9.7
0.060	180	4.2
	260	6.1
	340	7.9

Table 4.2. Room-temperature torsional strength of PY6/Ni/Incoloy 909 joints with various wall thickness and interlayer thickness.

Braze Alloys	Wall Thickness (in)	Interlayer Thickness (in)	Torque (in-lbs)	Alignment (in)	Failure
Au-Pd-Ni	0.015	0.062	160	0.041	in ceramic
			100	0.044	"
			125	0.050	"
	0.015	0.040	90	0.027	"
			130	-	"
Au-Pd-Ni (Phase I)	0.060	0.062	940	0.012	"
			700	0.012	"

4.2 Strength Evaluation for Various Materials Systems

Bond strengths of the joints made of PY6 silicon nitride and Inconel 600 were evaluated by room-temperature shear tests. Preliminary results showed that the shear strength of the joints with a Au-Ni-Cr-Mo-Fe alloy was in the range of 2-6.5 ksi, while a Au-Ni-Cr-Fe alloy provided strength levels in the range of 3-17 ksi. Intended bond area was 1 cm². Two new alloys exhibited excellent wetting (or bonding) between PY6 and Inconel 600 (50-100% bonding). The joint strength values were found to be a strong function of processing variables. Processing optimization with new alloys was needed to control the residual stress resulting from the high percentage of bonded area.

Thus, processing parameters were studied for the new braze alloys. Performance of the joints was consistently improved in terms of room-temperature shear strength for the PY6/Inconel 600 system as the SK-2 braze thickness increased (3-6 and 6-17 ksi for 0.002" and 0.004" thick foils, respectively). Also, it was noted that the pressure or weight applied to the samples during the brazing process significantly affected the bond strength.

This observation seems to be closely related to the nature of bonding attained from these new alloys and their ductility; adhesion of the joint was primarily due to the melting of the Au-rich phase. Therefore, the applied pressure and foil thickness have to be determined with discretion, depending on the properties of the braze alloys, i.e., melting phase, the gap between solidus and liquidus, and the viscosity of liquid metal.

Shear strengths of various joints were measured at room-temperature, 500°C, and 650°C. Joints were made between PY6 and one of the following substrate materials; Inconel 600, Incoloy 909, W, Mo, Ta, or Ni using one of the new braze alloys 0.002" thick. Similar joints were made with Au-Pd-Ni and Nioro (Au-Ni) as shown in Table 4.3. In general, the new alloys outperformed the conventional brazing alloys at high-temperatures. When these new alloys were used to bond ceramics to Ni or refractory metal substrates such as Mo, W, and Ta, the improvement in bond strength was significant. However, the performance with the new alloys at room-temperature was found to be low and somewhat inconsistent compared to those of conventional alloys, although it varies depending on the metal substrate used. These poor low-temperature strengths imply that at low-temperatures the joint strength controlled by the capability of the braze alloy and metal substrate to accommodate the residual stress. At high-temperature, the multiphase microstructure of the new braze alloys and the reduced residual stress synergistically enhance the joint strength. The braze alloys with single-phase microstructures like Au-Pd-Ni and Nioro, showed somewhat better performance at low-temperature, however they also showed minimal strength at high-temperature due to low creep resistance of these alloys. The PY6-Ni system was chosen since it can perform more or less consistently at room-temperature without oxidation of interlayer materials at high-temperatures. Evaluation of other interlayer materials such as Mo, W, and Ta, showed a significant oxidation problem when they were exposed at 650°C in air.

Table 4.3. Results of shear tests at various temperatures.

System	Brazing Alloy	Avg. Strength* (ksi)		
		RT	500°C	650°C
PY6† -Inc 600 -Mo -Ni	Au-Pd-Ni (ref.)	3.8	3.9	-
		9.3	21.0	-
		-	8.5	6.8
PY6 -Inc 600 (Zr)SNW -Ni -Mc -Ni	Nicro (ref.)	8.9	-	-
		8.9	11.6	-
		11.7	5.9	-
		8.2	13.5	12.3
PY6 -Ni -Mo -W -Ta -PY6 -Incoloy 909 -Inc 600	SK-1	8.6	14.2	14.5
		4.1	31.2	25.6
		5.1	-	10.5
		1.9	-	18.0
		-	-	7.5
		-	0	-
		2-6.5	-	4.0
PY6 -Ni -Mo -W -Ta -PY6 -Incoloy 909 -Inc 600	SK-2	5.4	15.2	11.3
		5.1	32.2	19.9
		-	34.2	33.9
		6.2	30.1	24.6
		-	-	6.9
		-	0.4	-
		3-17	4.0	6.4

* Strength values are an average of a minimum of 2 tests.

† PY6 was coated with 3 μ m Ti for wetting of brazing alloys.

5. PERFORMANCE TESTS OF CERAMIC-METAL JOINTS

5.1 Torsion Testing and Effect of Processing Conditions

A brazed joint between Incoloy 909 and PY6 is shown in Figure 5.1. The PY6 is on the left side, and the Incoloy 909 is on the right side. This type of joint was used for the torsion and torsion fatigue tests.

All of the mechanical testing of the brazed joints was carried out on a MTS model 646.25S hydraulic Axial/Torsion machine. The maximum torque of the machine is 20,000 in-lbs, and the maximum axial load is 55,000 lbs. The machine is equipped with hydraulic grips that can accommodate 0.5" diameter samples. The gripping pressure was adjusted to 1,500 psi to protect the ceramic from premature cracking in the gripped area during testing.

The torsion tests were run at a rate of 5-15 in-lb/sec in axial load and torque control modes. During the test, the axial load was maintained to within ± 1.0 lbs of zero. An induction furnace was used to heat the joints to 650°C for the elevated temperature tests. A SiC susceptor was used for more even heating of the ceramic-metal joint. The joints were equilibrated at the test temperature for 30 minutes before each test.

Room-temperature torsion tests were performed with cylindrical samples of 0.5" dia. x 4.0" long. Results are shown in Table 5.1. The average torsion strength of PY6 in Phase II was about 85% of previous results in Phase I.

2 of 2

Figure 5.1. Brazed joint between Incoloy 909 and PY6.

Table 5.1. Torsional strength of PY6 silicon nitride.

Material	Test Temperature (°C)	Torque (in-lbs)	Shear Strength (ksi)	Rotation	Diameter of gauge section (in)
Si ₃ N ₄ of Phase I	RT	2,110	86	0.40°	0.50
	650°C	525	171	1.84°	0.25
Si ₃ N ₄ of Phase II	RT	2,000	82	3.25	0.50
		1850	75	2.92	0.50
		1700	69	2.8	0.50

A total of 29 ceramic-metal joints were produced using SK-1 and SK-2 alloys which, in previous reports, were designated as B and C alloys, respectively. Alloy SK-1 has Mo in addition to Ni, Cr and Fe while Alloy SK-2 has Ni, Cr and Fe, as secondary elements (Table 3.16). These new high-temperature brazing alloys, especially SK-1, resulted in a significant gain in torsional strength at 650°C with a moderate strength loss at room-temperature. As shown in Table 5.2, the average joint strength made with SK-1 was 309 in-lb at 650°C whereas the strength at room-temperature was 380 in-lb. The 650°C torsional strength is far better than what we observed from the Au-Pd-Ni system of Phase I (10-60 in-lb, Table 2.4). It is to be noted that the new alloy maintained the room-temperature strength up to 650°C with little scatter in the failure strength values. However, the SK-2 alloy did not provide any gain in strength at low- and high-temperatures compared to those of Phase I. The lower strength values of joints brazed with the SK-1 and SK-2 alloys at room-temperature, as compared to the Au-Pd-Ni system, were attributed to high residual stress due to the low ductility of the SK-1 and SK-2 alloys.

For the production of PY6/Ni/Incoloy 909 joints, an optimum processing temperature was selected from the range of 1100°C to 1150°C on the basis of joint strength. As illustrated in Tables 5.2 through 5.4, performance at 650°C was improved with alloy SK-1 as the processing temperature decreased from 1150°C to 1100°C. The room-temperature strength of the joints made with SK-1 were to be less sensitive to the change in the processing temperatures. In contrast, the joints with SK-2 showed improved room-temperature torsional strength from 340 in-lb to 590 in-lb as the processing temperature increased. However, changing the processing conditions did not provide any noticeable improvement in high-temperature performance of the SK-2 braze. Of these three processing conditions, joints held at 1100°C for 30 minutes have resulted in the optimum performance for the SK-1 and SK-2 brazes.

Inferior performance of the joints at higher brazing temperature, i.e., 1150°C, was presumed to be related to the excessive phase separation and to the extent of interaction between Ti-coating and PY6 substrate. It was observed from the microstructural study that Alloys SK-1 and SK-2 consisted of Au- and Ni-rich solid solution phases. The Au-rich solid solution phase tends to segregate on the ceramic side from Ni-rich phase due to its low melting point, enabling the ceramic-metal to bond at relatively low temperature (<1150°C). Thus, high-temperature properties of the joints can be degraded due to the

increased amount of Au-rich phase in the vicinity of ceramic/braze interface as the brazing temperature increases. Excellent high-temperature performance of SK-1 could be attributed to the presence of a high-temperature strengthener such as Mo. It is expected that other solid solution strengtheners like Nb and V might provide the same effect as Mo. No significant difference in joint strength was observed at room-temperature from the joints made with SK-1 and SK-2. This indicates that both alloys have similar tensile properties which control the level of residual stress at the joints.

Table 5.2. Torsional strength of the PY6/Ni/Incoloy 909 system brazed with SK-1 or SK-2 braze alloy at 1100°C for 30 minutes.

Braze Alloy	Test Temperature (°C)	Torque (in-lbs)	Alignment (in)	Rotation (°)	Failure Mode
Au-Pd-Ni (Phase I)	RT	Avg. 540			in Ceramic
	650	Avg. 40			Slipped
SK-1	RT	405	0.003	1.23	in Ceramic
		420	0.008	1.30	"
		305	0.005	0.88	"
		310	0.006	---	
		390	0.006	---	
	650	307	0.015	1.23	in Ceramic
		290	0.019	0.95	"
		330	0.019	1.20	"
		145	0.014	---	>50% unbonded
SK-2	RT	400	0.020	1.25	in Ceramic
		400	-----	---	"
		315	0.005	0.92	"
		350	0.004	0.96	"
		240	0.002	---	"
	650	325	0.009	---	Failure in Ceramic
		15	0.005	---	>50% unbonded
		90	-----	---	Slipped

Table 5.3. Torsional strength of the PY6/Ni/Incoloy 909 system brazed at 1125°C for 20 minutes.

Braze Alloy	Test Temperature (°C)	Torque (in-lbs)	Alignment (in)	Rotation (°)	Failure Mode
SK-1	650	220	0.014	2.5	Ceramic
SK-2	650	68	0.026	0.25	Slipped

Table 5.4. Torsional strength of the PY6/Ni/Incoloy 909 system brazed at 1150°C for 30 minutes.

Braze Alloy	Test Temperature (°C)	Torque (in-lbs)	Alignment (in)	Rotation (°)	Failure Mode
SK-1	RT	400	0.014	1.15	Ceramic
		440	0.010	1.25	
	650	340	0.010	1.20	Slipped
		72	0.015	0.37	
	750	40	0.017	1.15	Slipped
SK-2	RT	475	-----	-----	
		650	0.014	2.25	
		640	0.015	1.87	
	650	10	0.018	-----	
		75	0.028	0.27	
		50	-----	0.15	

5.2 Torsional Creep Testing

From Phase I of the program, it was found that the joints made with Au-Pd-Ni were not able to sustain any significant load (>20 in-lb) at 650°C even though they performed successfully at room-temperature and 500°C. Therefore, the main goal of this alloy development is to produce ceramic-metal joints which can survive at 650°C without a significant loss of room-temperature performance. The Au-Pd-Ni alloy of Phase I provided virtually no resistance to creep at this temperature because to the microstructure consisted of a single solid solution phase.

Table 5.5 summarizes the creep test result of SK-1 joints at 650°C. At 185 in-lb torque level, the rupture lives of four joints were 3.5 hours, 10.5 hours, >100 hours, and >160 hours, respectively. Testing of the first two samples was completed by rupture failure at the interface between braze and PY6. These samples showed a high portion of unbonded area (>30%) in the joints. Testing of the other two joints were stopped after 100 hours and 160 hours of creep without failure. The performance of these joints at 650°C was outstanding. Excellent performances were expected as long as the bonded area exceeds more than 80% of the intended area.

Table 5.5. Torsional rupture of PY6/Ni/Incoloy 909 joints at 650°C.

Braze Alloy	Constant Torque (in-lbs)	Alignment (in)	Rotation (°)	Time to Rupture (hrs)	Creep Rate (°/hr)
SK-1	185	0.034	0.58	3.5	4.5×10^{-2}
	185	0.022	1.265	10.5	6.7×10^{-2}
	185	0.025	0.640	>100*	8.8×10^{-4}
	185	0.008	1.317	>160 (1 week)	4.6×10^{-3}

* Experiment was stopped after 100 hours creep at 650°C due to a planned electrical shut-down.

The first sample showed the creep rate of $0.045^{\circ}/\text{hr}$, and failed when it rotated about 0.22° more from the end point of the first stage creep (0.50°). The steady state creep of the third sample started from 0.552° and stopped at 0.640° in 100 hours, resulting in the creep rate of $0.00088^{\circ}/\text{hr}$. A significant difference in creep rate must be related to the quality of the joined area, i.e., the percentage of unbonded area and its distribution.

Figure 5.2 is a plot of creep behavior of the fourth sample which survived more than 160 hours (1 week) at 650°C . The average creep rate of the second stage was $0.0046^{\circ}/\text{hr}$, which is five times higher than that of the third sample. If this creep rate can be used for the third sample, the useful life of this sample would be more than 800 hours. This result in creep life was quite significant for the future application of this type of joints since the expected life of an automotive engine is about 3000 hours.

A significant difference in the creep rates were recorded during the second stage of creep. Changes in the creep rate were always associated with temperature fluctuation from 620°C to 670°C . This implies that a small change in use-temperature could cause a significant effect on the rupture lives of the joints in this temperature range. The heating was done by an induction heating unit. As indicated in Table 5.5, the creep rate was not related directly to the rupture life of the joint. The rates seemed to be strongly controlled by the distribution of unbonded area.

Excellent high-temperature creep performance was due to the dual phase structure of SK-1 alloy. Most deformations are expected to occur in the Au-Ni-Cr phase at high-temperature, while the Ni-Cr-Fe phase gives rise to obstruction to the flow of the Au-rich phase. However, the existence of two different phases of different melting points entails inconsistency in bonded area after brazing process. There is a need to develop a better processing technique or modified alloys to ensure the consistent bonded area to improve the reliability of the joints.

Figure 5.2. 650°C creep behavior of ceramic-metal joints brazed with the SK-1 alloy.

5.3 Fatigue Testing

5.3.1 Thermal Fatigue of Brazed Joints

The geometry and mass of a gas turbine rotor play a large part in determining the internal temperatures of the rotor/shaft system; therefore, one system was chosen as a model. Thermal maps of the ceramic turbine rotor and structural alloy shaft cross-section of the chosen model were based on work done at General Motors on their gas turbine engine as shown in Figure 5.3.

The location of the 650°C isotherm was marked on the thermal map for steady state, maximum power conditions for both materials. This area was then examined on the thermal maps for steady state idle conditions. The idle temperatures were found to be 335°C and 465°C for the Si_3N_4 . The Si_3N_4 required approximately 130 seconds to reach 650°C from 465°C and return again. Figure 5.4 is a graphical representation of the thermal cycle used for testing.

Figure 5.3. Temperature profile in the vicinity of the turbine rotors.

Figure 5.4. Thermal cycle used in thermal fatigue tests of brazed joints for a 650°C use temperature.

5.3.2 Mechanical Fatigue of Brazed Joints

The mechanical fatigue tests were run under torque control along with axial load controlled to within ± 1.0 lbs of zero throughout the testing. The tests were run between 35 and 185 in-lbs, with a mean torque level of 110 in-lbs, which represented actual conditions in an automotive engine during idling and at maximum speed (rpm). A loading frequency in the range of 0.016-1.5 Hz was used for room-temperature fatigue testing (Figure 5.5). Two or three joints were fatigue tested at room-temperature, 600°C, and 650°C.

Figure 5.5. Mechanical cycle used for room-temperature fatigue testing of the brazed joints.

All of these joints tested at room-temperature easily met the test requirements and any further testing was felt to be unnecessary. Therefore, two joints were tested at room-temperature as shown in Table 5.6. The room-temperature fatigue properties of SK-1 joints were excellent, far exceeding the specified number of cycles (1000 cycles) at this torque amplitude. However, the joint made of SK-2 did not survive a single cycle, but failed during loading.

For high-temperature fatigue testing, testing parameters such as torque loading rate, i.e., 10-15 seconds to reach to the full speed, and duration time at

maximum and minimum torque values, 10-20 seconds, were determined arbitrarily based on the commercial information for an auto performance and effectiveness of the data acquisition.

Two mechanical fatigue tests were performed at 600°C and failed at in the vicinity of 135 in-lb torque while loading. These samples were found to have more than 50% unbonded area. For these tests, a 0.016 Hz loading frequency was used with 10-20 seconds of duration at maximum or minimum torque values. The loading rate was 10-15 in-lb/sec. which, in turn, takes 10-15 seconds to reach a full speed in practical application. Further evaluation with proper joints is needed for this experiments.

Also, a SK-1 joint tested at 650°C failed during spanning the torque amplitude from 110 in-lb torque. The frequencies of fatigue tests were 1.5 and 0.04 Hz for room-temperature and 650°C, respectively, without holding time at maximum or minimum torque values. The low frequency used for the fatigue test at 650°C was again to simulate the acceleration rate of an engine, roughly 12.5 seconds from zero to full speed. It was speculated for SK-1 joints, besides the influence of the high portion of unbonded area, that the repeated cycling might accelerate the second stage creep rate at high-temperature, causing premature failure.

Table 5.6. Torsional fatigue of the PY6 /Ni/Incoloy 909 system.

Braze Alloys	Test Temperature (°C)	Torque Amplitude (in-lbs)	Alignment (in)	Rotation (°)	Number of Cycles for Failure
Au-Pd-Ni (ref.)	RT	35-185	-----	---	>1,000,000
		70-370	-----	---	>1,000,000
SK-1	RT	35-185	0.016	0.59	427,440
		35-185	0.020	0.51	>1,000,000
	650	35-185	0.025	---	Loading Failure
SK-2	RT	35-185	0.026	---	Loading Failure

6. SUMMARY AND CONCLUSIONS

Finite element analysis and probabilistic failure analysis were used to identify important concepts in the design of ceramic-metal brazed joints.

An interlayer was found to be necessary to prevent fracture of the ceramic during the cooling down period after the brazing process. The cup geometry with an interlayer resulted in the highest probability of survival.

This study showed that interlayers were effective in reducing the residual stresses in the ceramic. For joints between Si_3N_4 and typical metal superalloys at room-temperature the ideal braze mechanical properties, in order of importance, are low flow stress, high thermal expansion, and a low Young's modulus. The ideal interlayer properties, in order of importance, are a low flow stress and a high thermal expansion, and the ideal structural alloy has a low coefficient of thermal expansion.

PY6 was selected again for Phase II of this program over SNW1000 and AY6 Si_3N_4 because of its excellent high-temperature strength and its compatibility with the coating material selected. Incoloy 909, the low-expansion Fe-based superalloy, was the structural alloy of choice. Due to a concern over the effect of the high-temperature brazing process ($>1000^\circ\text{C}$) on the mechanical properties of Incoloy 909, a post-heat treatment procedure was developed to regain the strength of Incoloy 909.

Room-temperature and 500°C torsion strengths of the 650°C system were measured in the range of 30-100 N-m with a 2 cm^2 brazed area while the strength at 650°C was significantly lower (1.6-7.0 N-m). This was attributed to a reduction in the shrink-fit at 650°C and a loss in strength of the braze alloy. The $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ joints showed excellent room-temperature fatigue behavior. A similar trend was seen in the high-temperature strength of the $\text{Si}_3\text{N}_4/\text{Mo}/\text{Inconel 718}$ joints, which had lower strength than the $\text{Si}_3\text{N}_4/\text{Ni}/\text{Incoloy 909}$ joints due to the high coefficient of thermal expansion of Inconel 718.

The strength predictions of the finite element analysis were compared with experimental results. Scatter in the measured strengths and the difference between measured and predicted strengths indicated the importance of processing effects and the probabilistic nature of ceramic failure on the fracture process. Fractographs of room-temperature torsion specimens showed that the initiation sites for cracking coincided with those

for maximum principal stress. This indicated that the maximum principal stress criterion should be a good estimate of the location of probable fracture. However, the probabilistic approach for the ceramic-metal brazed joints was a better method for comparing the performance of various brazed joints due to the statistical nature of ceramic failure, and it provided an estimate of the strength distribution of the joint for design considerations.

A new bimaterial failure theory was developed and the predictions from this theory were compared to experimental joints. This new theory results in a reduction in the stress in the ceramic near the ceramic/braze interface due to plastic deformation of the braze layer. Therefore, the predicted joint strength increases when the braze plasticity is considered.

It is expected for the ceramic-metal joints that the joints can maintain not only desirable room-temperature performance but high-temperature strength, hot corrosion resistance, and creep strength. However, there is no brazing alloy designed specifically to meet these requirements of ceramic-metal joints. In order to develop high temperature brazing alloys for ceramic-metal joints, two approaches were taken with Ni-based or Au-based alloys with emphasis on either (1) brazability of ceramic-metal or (2) high-temperature performance.

Ni-based alloys were developed to meet the required brazing temperature by introducing metalloids into the system. They have shown unacceptable losses of ductility due to the segregation of the metalloids on the grain boundary. Meanwhile, additions of IB elements (up to 20%) to Ni matrix were not enough to reduce the brazing temperature below 1150°C. The Au-Ni-Cr-Mo-Fe and Au-Ni-Cr-Fe alloys which melt at 1050-1100°C have met the processing requirements for high temperature ceramic-metal joints. Thus, these two alloys were chosen and evaluated in terms of microstructure, joint strength, and creep strength.

Microstructures of the joints made of Au-Ni-Cr-Mo-Fe (SK-1) or Au-Ni-Cr-Fe (SK-2) alloys showed two discrete Au- or Ni-based phases. The bonding between silicon nitride and Ni interlayer was found to rely on the spreading of low melting phase, the Au-based phase, at the ceramic/metal interface. Also, the thin layer of Au phase at the interface could start deforming at a much lower stress level, providing better room-temperature performance. High-temperature creep resistance was obtained from the Ni phase and the discontinuous Au-phase. Braze foil production of two alloy compositions, Au-

Ni-Cr-Mo-Fe and Au-Ni-Cr-Fe alloys, was completed. The cast bar was rolled into 0.002" thick foil. The successful rolling of this alloy was attributed to its excellent toughness and ductility.

It was found that the high-temperature brazing alloys, especially SK-1, resulted in a significant gain in the torsional strength of the joints at 650°C with a moderate loss at room temperature. The average joint strength made of SK-1 was 309 in-lb at 650 °C whereas the strength at room temperature was 380 in-lb. The high temperature torsional strength is far better than what we observed from the Au-Pd-Ni system of Phase I (10-60 in-lb). The new alloy seems to maintain the room-temperature strength up to 650°C with little scatter in the failure strength values. Alloy SK-2 did not provide much gain in strength at low and high temperatures compared to those of Phase I. It needs more investigation in processing technique for the property optimization of the SK-2 joints. Lower strength values with new brazing alloys at room temperature than that of the Au-Pd-Ni system (avg. 540 in-lb) were due to high residual stress resulted from the lower ductility of these new alloys.

The rupture lives of four joints made of new brazes were 3.5 hours, 10.5 hours, >100 hours, and >160 hours, respectively, at 185 in-lb torque level. Most rupture failures occurred at the interface between braze and PY6. The samples of short rupture lives showed a high portion of unbonded area (>30%) in the joints. The performance of these joints at 650°C was outstanding compared to other ceramic-metal joints produced with any existing braze alloys. Excellent performances were expected when the bonded area exceeds more than 80% of the intended area.

This significant improvement in creep performance was due to the dual phase microstructure of SK-1 alloy. Most deformations are expected to occur in the Au-Ni-Cr phase at high temperature, while the Ni-Cr-Fe phase gives rise to obstruction to the flow of the Au-rich phase. However, the existence of two different phases of different melting points entails inconsistency in bonded area after brazing process. There is a need to develop a better processing technique or modified alloys to ensure the consistent bonded area to improve the reliability of the joints.

In addition, the ceramic-metal joints produced using Alloys SK-1 and SK-2 were subjected to 1000 thermal cycles. These joints were examined for the development of cracks in the ceramic part nondestructively by a microfocus X-ray. It was found that none of the joints showed any evidence of

cracking, implying that the thermal fatigue did not cause any cracking or the sizes of cracks were under the limit of x-ray detectability which is about $1\mu\text{m}$. Also, excellent room-temperature fatigue properties were noted from the SK-1 joints at the fatigue amplitude of 35-185 in-lbs., while the fatigue performance of the joints at 650°C was not determined due to the lack of samples.

Overall results clearly showed that the statistical approach to design and evaluate the geometries for ceramic-metal joints was effective and that new high temperature brazing alloys had excellent potential to be used for heat engine or other high-temperature applications. Further refinement in alloy compositions and optimization of processing conditions are recommended for commercialization of these alloys.

PHASE II STATEMENT OF WORK

Phase II. Material System and Design Optimization, and Joint Life Prediction

The goal of Phase II is to optimize materials systems and joint designs building on the results of Phase I. The work shall also include demonstration of the potential for scale-up of the joint size to interfacial areas of commercial significance, applicability of the analytical joint modeling tools and the ability to use these tools to design and predict the mechanical and thermal stability of the larger joints. These joints, referred to as "scale-up" joints, shall have the ceramic shaft at least 0.8 inches in diameter. The goal is to develop a system which can perform in an engine such as the ATTAP engine. The anticipated environment will be oxidizing and will have joint temperatures of up to 950°C. Improved joint materials systems shall be developed to optimize the combination of competing properties which include ductility, yield strength, and creep resistance. The effect of each of these properties on joint performance shall be examined using the FEM model. Finally, a program of mechanical testing shall be carried out to confirm the effectiveness of the modeling program. This shall include torsion tests, thermal and mechanical fatigue, and creep testing. In addition, an analytical and experimental program in service life prediction will be undertaken. The work in Phase II shall include five tasks (Tasks 5, 6, 7, 8 and 9):

Task 5. Materials System Optimization

In Phase I of this project, Inconel 718 and Incoloy 909 were selected as the structural alloys, nickel and molybdenum were selected as the interlayer materials, and PY6 (silicon nitride - 6 wt. % yttria) was shown to be the preferred ceramic. The continued program will be centered around these materials. Several braze alloys based on the Au-Pd-Ni and Au-Pd systems were studied. Because of the high-temperature strength of the structural materials and the joint geometry, the braze alloy is expected to be the weakest mechanical link in the joint system. Therefore, there is a need for the development of new braze alloys to provide the improved high-temperature properties required for the 650°C and 950°C ceramic-to-metal joints.

5.1. Braze Alloys

The starting point for braze alloy development will center on defining the limiting properties of a successful braze alloy. The FEM model developed during Phase I of this project will be used to define the amount of plastic deformation carried by the interlayer and braze materials. FEM will be used to determine the minimum amount of deformation in the braze alloy that is required to obtain a crack-free joint. The sensitivity of a successful, crack-free joint on the plasticity and yield strength of the braze alloy will also be determined. Once the limiting mechanical requirements of the braze alloy are known, candidate material systems for the braze alloy will be identified and studied.

The first group of brazing alloys to be studied will be based on metal eutectic compositions with sufficient ductility and high-temperature oxidation resistance. Compositions of this type will promote fluidity and minimize porosity due to shrinkage during solidification. High melting point, oxidation-resistant metal systems will be evaluated with emphasis on metal eutectic systems (in contrast to non-eutectic Au-Ni or Pd-Ni) and on Ni-based alloys. The principle of diffusion solidification will be taken into consideration in the design of braze alloys.

A second group of candidate brazing alloys will be evaluated concurrently with the above group. These will be based on superalloy compositions and turbine blade repair braze alloys. Their relatively high yield stress and low ductility (compared with precious metals) should make them less forgiving and require reliance on the interlayer to accommodate differential expansion. The FEM model will be used to establish whether such a set of properties is a viable approach.

Initial screening of potential alloy systems will be based on published phase diagrams. Five samples will be required in each system to establish wetting. It will also be necessary to measure the mechanical properties of the braze with a minimum of three tensile specimens for each composition. The interactions between the braze alloy and metals will be examined using coupon samples. About twenty per composition are anticipated.

5.2. Interlayers

Previous work conducted in Phase I demonstrated the relative effectiveness of low yield strength materials and low CTE materials as interlayers in reducing the joint stress. Typical interlayer materials emphasized in Phase I were pure materials such as nickel, copper, molybdenum, tungsten and niobium. Improvements in performance are possible by using alloys which could provide combinations of properties unobtainable in pure materials. Service temperatures of the joints require combinations of high-temperature properties which are possible through alloying. For example, a nickel alloy could provide adequately low yield stress to avoid cracking the ceramic, while having improved creep resistance compared with pure nickel. Refractory metal alloys like Mo- and W-base could be acceptable with improved ductility, retaining their high-temperature properties and low CTE. Various alloys will be examined based on the working model developed in Phase I.

New interlayer ideas, such as multilayer materials, will also be examined in Phase II. An example of a multilayer material is an outer ring of Mo with an inner ring of Ni. Preliminary FEM conducted in Phase I show promise for these types of interlayers. The planned work would screen three or four candidate interlayers based on yield strength, CTE, creep strength, and oxidation resistance. Performance tests of room-temperature and creep strength will be carried out. Published data will be used to verify oxidation resistance and CTE.

5.3. Coatings

The study on new braze alloys and new interlayer materials will necessitate a review of the materials used to coat the ceramic component. Coatings on the ceramic are necessary in order to obtain wetting and adhesion between the chemically dissimilar braze and ceramic. The adhesion or wetting of a metal to a ceramic depends on the chemical compatibility between the metal and ceramic. Included in the concept of chemical compatibility is the nature of the wetting atmosphere, chemical bonds, chemical reactions, surface energy of the ceramic, and surface tension of the metal. The methodology for testing coatings developed in Phase I will be applied to the new study.

Task 6. Joint Design Optimization

6.1. Development of multiple interlayer joints (with Brown U.).

Various multilayer systems shall be examined to minimize the stress generated from thermal expansion mismatch at the ceramic-metal interface. Analytical expressions shall be modified to account for the introduction of multiple interlayers in the structure. The modified expressions shall be used for the materials systems suggested by Task 5 with the use of finite element methodology.

6.2. Stress analysis of brazed joint and optimization (with Brown U.).

Stress analysis shall be performed to take into account the dimensional change and composition change caused by the brazing process. FEM analysis of the joint system shall be done using the geometry of the joint and the properties of the braze alloy after brazing. Numerical stress analysis using the commercially available code, ABAQUS, shall be performed. The cylindrical joint shall be the basic joint design for consideration.

6.3. Stress analysis of structural alloy and optimization (with Brown U.).

The effect of the overall macroscopic joint shape on the residual stress in the ceramic-metal brazed joint shall be studied by examining axisymmetric joints other than simple cylinders. The effects of tapers and reduced wall thickness are two of the geometrical factors that shall be addressed. The geometry of the structural alloy shaft shall be changed to reflect the findings.

Task 7. Joint Life Prediction Methodology and Testing

7.1. The gamma-criterion developed in Phase I shall be improved (with Brown University).

Material data on the damage assessment and the crack growth, particularly for the ceramic components, shall be collected. Modeling of the crack growth failure mechanism shall be developed as a part of the ABAQUS-supported finite element braze joint model.

7.2. Mechanical fatigue.

Mechanical fatigue tests shall be performed on the MTS machine

purchased during Phase I. These tests shall cycle between 35 and 185 in-lbs with a frequency of 1.5 Hz. Ten samples (2 cm² joint area) shall be tested at each of two temperatures, 650°C and up to 950°C, for a total of twenty samples. Mechanical fatigue tests shall be run at constant temperature. The superimposed effects of mechanical and thermal cyclical loading may be investigated if time permits.

7.3. Creep characteristics of brazed joints.

The creep characteristics of the final brazed joint shall be characterized. A brazed sample (2 cm² joint area) shall be loaded at a constant torque of 185 in-lbs and the rotational strain shall be measured as a function of time. These tests shall be carried out at 650°C and another temperature, up to 950°C. Ten samples shall be tested at each temperature, for a total of 20 samples.

Task 8. Fabrication and Evaluation of Scale-Up Size Ceramic-to-Metal Joints

8.1. Scale-Up Brazed Joint Production

Brazed samples shall be produced for performance testing. Joint design shall be based on analytical and experimental results from earlier portions of the program. These joints shall be prototypical of rotor-to-shaft joints in the ATTAP program. The ceramic shaft end in this engine has a diameter of about 0.8 inches. The length of the brazed ring around this shall be determined using FEM to evaluate the relationship between two competing effects. If the brazed length is excessively long, the likelihood of cracking the ceramic is high, resulting in weak joints. If the joint is too short, the stress in the braze would probably exceed its capacity. The FEM model shall be used to yield a workable solution and minimize the number of experimental samples needed. A minimum of 3 test joints shall be fabricated and tested, each at room-temperature, 650°C and 950°C.

Task 9. Reporting and Quality Assurance Requirements

Reporting and quality assurance requirements will be identical to those used for Phase I.

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APPENDIX A - MATERIALS PROPERTIES

In order to express the response of the joint as a function of material properties and temperature, constitutive equations were developed for each joint material. The joint materials studied were; silicon nitride ($\text{Si}_3\text{N}_4 + 6 \text{ wt}\% \text{Y}_2\text{O}_3$), a Au-5Pd-2Ni braze alloy, an Incoloy 909 metal shaft, an Inconel 718, and a nickel 200 or molybdenum MT-104 interlayer.

The uniaxial stress and strain relations of the materials were considered to be temperature-dependent, but rate-independent. It was assumed that the effect of heat generation due to plastic work on constitutive models was negligible because of the comparatively large heating process during brazing of the joint. The silicon nitride was treated as a perfectly elastic material.

The coefficients of thermal expansion (CTE) and the mechanical properties of the materials in the joint are given in Figure A-1 and Tables A-1, A-2, and A-3. The temperature dependent Young's modulus data for the braze alloy, Incoloy 909, Inconel 718, and the silicon nitride were approximated by the following equation:

$$E(T) = E^* \left(1 - a \frac{T}{T_m} \right), \quad \text{Eq. A1}$$

where E^* is the extrapolated Young's modulus at absolute zero degrees Kelvin and T_m is the melting temperature of the material in degrees Kelvin. The empirical constant "a" was 0.33 for covalently bonded ceramics and 0.47 for metals. For the braze alloy the modulus was derived using a room-temperature modulus of 92 GPa and T_m of 1343°K, for Incoloy 909 and Inconel 718 158 GPa and T_m of 1673°K were used [14], and for silicon nitride 296.5 GPa and T_m of 2173°K were used.

The stresses in the elastic region were described by the following equation for all materials:

$$\sigma = E(T)\epsilon \quad \text{Eq. A2}$$

where σ is true stress, ϵ is true strain, and $E(T)$ is Young's modulus as a function of temperature. The plastic portion of the stress strain curve for the

nickel and molybdenum interlayers was described by the following equation;

$$\sigma(T, \epsilon) = \sigma_y(T) + [\sigma_u(T) - \sigma_y(T)] \left(\frac{\epsilon}{\epsilon_f(T)} \right) \quad \text{Eq. A3}$$

where $\sigma(T, \epsilon)$ is the stress at a given temperature (T) and plastic strain (ϵ), σ_y is the yield stress (or flow stress), σ_u is the ultimate tensile strength, and ϵ_f is the strain at failure. Equation A3 amounted to a straight line approximation of the plastic region of the stress-strain curve.

The stress-strain data for the Incoloy 909 was calculated by the following equation:

$$\sigma = f(T)\epsilon^n \quad \text{and} \quad f(T) = aT + b \quad \text{Eq. A4}$$

where n is the strain hardening coefficient and $f(T)$ was a temperature dependent strength coefficient. The constants " n ", " a ", and " b " used for Incoloy 909 were 0.0575, -0.3478 MPa/°C, and 1338 MPa, respectively.

Temperature dependent Poisson's ratios used for Incoloy 909, Inconel 718, and molybdenum are listed in Table A-3 and Table A-4. Poisson's ratios for the braze alloy, silicon nitride, and nickel 200 were treated as constants due to lack of temperature dependent information, they were taken as 0.36, 0.23, and 0.31, respectively.

Table A-1. Stress-strain data for the Au-5Pd-2Ni braze alloy tested at 20°C, 650°C and 800°C.

20°C Tensile Tests			650°C Tensile Tests			800°C Tensile Test	
True Strain	Stress (MPa)		True Strain	Stress (MPa)		True Strain	Stress (MPa)
	Test #1	Test #2		Test #1	Test #2		
0.000	121.6	108.6	0.000	46.27	48.92	0.000	32.16
0.002	127.6	111.9	0.004	49.70	52.89	0.005	38.60
0.004	130.9	115.2	0.008	52.22	54.87	0.012	42.18
0.006	132.9	116.9	0.013	53.86	56.51	0.018	44.44
0.008	134.8	118.5	0.017	55.52	57.52	0.025	46.43
0.010	136.2	120.1	0.021	56.85	58.83	0.032	47.49
0.014	138.8	121.8	0.025	57.51	59.50	0.038	48.62
0.018	140.4	125.3	0.033	59.47	60.81	0.045	49.61
0.022	142.1	127.8	0.042	60.14	61.48	0.052	50.47
0.026	143.5	130.0	0.050	61.15	62.59	0.059	51.25
0.030	145.4	131.7	0.058	61.48	63.13	0.065	51.96
0.033	146.7	133.3	0.066	62.12	63.46	0.072	52.61
0.037	148.1	135.0	0.075	62.79	63.80	0.079	53.21
0.075	159.3	154.7	0.083	63.13	64.10	0.086	53.76
0.112	169.2	174.5	0.091	63.46	64.14	0.093	54.28
0.147	177.8	185.3	0.100	63.60	64.20	0.106	55.24
0.181	186.4	---					

Table A-2. Tensile properties of nickel 200 as a function of temperature [15].

Temperature °C	Flow Stress, σ_y (MPa)	Ultimate Tensile Strength, σ_u (MPa)	Elongation at Failure, ϵ_f	Young's Modulus (GPa)
20	149	459	0.473	211
100	149	456	0.452	207
200	143	466	0.445	203
300	132	458	0.460	198
400	117	341	0.633	192
500	98	242	0.660	186
600	79	170	0.724	180
700	59	119	0.826	174
800	41	83	0.965	167
900	26	58	1.141	160
1000	14	38	1.991	153
1100	9	19	2.054	145
1200	3	5	2.117	137

Table A-3. Tensile properties of molybdenum MT-104 as a function of temperature [16,17].

Temperature °C	Flow Stress, σ_y (MPa)	Ultimate Tensile Strength, σ_u (MPa)	Elongation at Failure, ϵ_f	Young's Modulus (GPa)	Poisson's Ratio
20	665	777	0.16	320	0.308
100	627	714	0.148	316	0.306
200	590	652	0.135	312	0.306
300	559	604	0.121	308	0.306
400	535	569	0.114	303	0.307
500	515	542	0.106	299	0.308
600	498	522	0.10	294	0.312
700	482	505	0.10	290	0.316
800	466	489	0.10	286	0.321
900	447	471	0.10	281	0.327
1000	425	446	0.10	277	0.334
1100	397	414	0.09	273	0.342
1200	361	371	0.12	269	0.350

Table A-4. Tensile properties of Inconel 718 as a function of temperature [18].

Temperature °C	Flow Stress, σ_y (MPa)	Ultimate Tensile Strength, σ_u (MPa)	Elongation at Failure, ϵ_f	Poisson's Ratio*, ν
20	1140	1350	0.26	0.346
100	1065	1315	0.21	0.341
200	1065	1260	0.19	0.339
300	1065	1245	0.17	0.339
400	1065	1245	0.17	0.342
500	1030	1210	0.16	0.347
600	995	1155	0.16	0.354
700	925	1030	0.10	0.363
750	833	925	0.06	0.368
800	745	820	0.08	0.374

* these values were used for Inconel 718 and Incoloy 909.

Table A-5. Properties used for the nickel aluminide braze alloy [5]. Poisson's ratio was kept constant at 0.3 and the CTE varied linearly from $10 \times 10^{-6}/^{\circ}\text{C}$ at 20°C to $15 \times 10^{-6}/^{\circ}\text{C}$ at 1000°C .

Temperature ($^{\circ}\text{C}$)	Young's Modulus (GPa)	True Strain	True Stress (MPa)
20	150	0.0	500
		0.01	833
		0.02	1042
		0.03	1250
327	265	0.0	458
		0.01	833
		0.02	938
		0.03	1063
527	290	0.0	375
		0.01	667
		0.02	833
		0.03	917
727	190	0.0	208
		0.01	333
		0.02	333
		0.03	354

Figure A-1. Coefficients of thermal expansion for the materials used in the ceramic-metal joints. Nickel, molybdenum, Au-5Pd-2Ni [19], Incoloy 909 and Inconel 718 [18], and silicon nitride [20]. Data for gold was used for the Au-5Pd-2Ni alloy.

**APPENDIX B - CERAMIC-FAILURE MODEL FOR BIMATERIAL
SYSTEMS - THEORY**

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