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**Tensile Strength As a Function of
Thermal History of Inconel 718 and
Inconel 625 Alloys for
Glass-Ceramic Headers**

Michael C. Rey and William R. Henderson

June 11, 1982

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Abstract

Tensile strength tests were conducted on Inconel 718 specimens following a variety of heat treatments, and on as-received and heat-treated specimens of Inconel 625. A heat treatment cycle for Inconel 718 was found that represents an acceptable compromise between a thermal cycle that yields the strongest metal and one that least taxes a glass-ceramic material to which the Inconel 718 is bonded. Heat treating resulted in a moderate decrease in the tensile strength of the as-received Inconel 625.

Introduction

Inconel 718 is a nickel-based superalloy [1] that is corrosion resistant and age hardenable. For these reasons Inconel 718 is being considered as a housing material for a glass-ceramic header. In the course of fabricating the header, the metal housing, as well as the electrical feed-through pins that comprise the other metal parts of the header, undergoes a heat treatment required to bond the housing and pins to the enclosed mass of glass and crystallize the glass*. Because an unadulterated sealing and crystallizing cycle would leave the Inconel 718 considerably less strong than is desirable, an age hardening heat treatment is included as part of the total heating cycle. To optimize the mechanical strength of Inconel 718, a precipitation hardening cycle is recommended by the ASTM [2]. However, the long holds required at the hardening temperatures specified in the standard cycle result in undesirable evolutions of the glass-ceramic microstructure with attendant degradation of the glass-ceramic properties. These effects on the glass-ceramic have been characterized by microscopic examination

of the heat-treated glass-ceramic, by x-ray diffraction, and by dilatometry. Their principal manifestation in the header is the fracturing of the bonded glass-ceramic, which renders the header unserviceable. The problem addressed in this report is that of modifying the sealing-crystallizing-hardening cycle so that the glass-ceramic is not adversely affected while the Inconel 718 is adequately, if not optimally, strengthened.

Inconel 625, another nickel-based superalloy [1], is considered suitable for use in glass-ceramic headers, although this alloy is not age hardenable. Inconel 625 is included in this study to obtain a direct comparison between its tensile properties and those of Inconel 718.

Experimental procedures

As-received rods of Inconel 718 and Inconel 625 (Metalmart, Inc., Whittier, California) were machined into tensile specimens with a gauge length of $0.750 \text{ in.} \pm 0.005 \text{ in.}$ and a gauge diameter of $0.160 \text{ in.} \pm 0.003 \text{ in.}$ The overall specimen shape conformed to ASTM specifications [3]. The machined specimens were ultrasonically cleaned in four sequential baths: methylene chloride, detergent, distilled water, and isopropyl alcohol. The cleaning was necessary to

*S-glass, Sandia National Laboratories, Albuquerque, New Mexico.

prevent contaminating the furnace in which the heat treatments were carried out. The specimens were grouped into batches of five each and heat treated (or not) according to the schedules listed in Table 1. All heat treatments were carried out in argon at one atmosphere of pressure. Rockwell hardness was measured, then the specimens were tensile tested in an Instron mechanical testing machine using an elongation rate of 0.05 in./min. Specimen microstructures were examined by means of optical and scanning electron microscopy.

In the heat treatment schedules shown in Table 1, the slow cooling rate at the end of each cycle served to anneal and to prevent the thermal shocking of the glass-ceramic to which the metal alloy would be bonded in the course of device fabrication. Batch 718-4 was precipitation-hardened before undergoing the sealing cycle. The purpose was to determine how much additional strength would be left in a previously hardened alloy after it experienced the sealing cycle.

Results and discussion

The results of the hardness and tensile tests are shown in Table 2. The hardness of the as-received Inconel 718 was measured on the Rockwell N scale, and the number was converted to the C scale number shown. It is clear from the data that heat treating Inconel 718 increases its hardness and tensile strength significantly. Batch 718-4 is much weaker than the other heat treated batches; however, the sealing cycle did not completely homogenize the alloy to a degree approaching the as-received condition. Batches 718-5 and 718-6 both exhibit good tensile

strength, but the total heat treatment of batch 718-6 required only 7 hr while batch 718-5 underwent a 17 hr cycle. For minimizing the time at elevated temperatures for the glass-ceramic in the header, the heating schedule for batch 718-6 is preferred. Dilatometer data for glass-ceramic specimens subjected to this cycle have fallen within described limits, and headers have been successfully fabricated (i.e., without fracturing the glass-ceramic), using the heating cycle of batch 718-6.

Optical micrographs of specimens etched in HCl from batches 718-1 and 718-6 are shown in Figure 1. Although the two specimens have very different thermal histories, and one is much stronger than the other, no striking differences in their microstructures are apparent. The precipitates responsible for hardening are far too small to be imaged optically, and a transmission electron microscope was not available for this study. The precipitation hardening process in Inconel 718 is, however, well understood [4,5]. The primary hardening constituent is the gamma double prime phase of Ni_3Cb . Other precipitated phases in heat-treated Inconel 718 include a $(\text{Cb}, \text{Ti}, \text{Al})\text{C}$ phase within the grains and an intergranular Laves phase identified as $(\text{Ni}, \text{Fe}, \text{Cr})_2(\text{Cb}, \text{Ti}, \text{Mo})$. With the use of a transmission electron microscope, tensile strength can be correlated with the distribution of fine precipitates.

The test results for the Inconel 625 specimens indicate that the heat treatment was equivalent to an anneal of this alloy. The fact that the tensile strength was not greatly lowered by the heat treatment indicates that the as-received Inconel 625

Table 1 - HEATING TREATING SCHEDULES

Batch No.	Heat Treatment
718-1	As received.
718-2	<p>"Standard" cycle. Heat from room temperature to 1000°C at 100°C/min; hold for 8 min. Cool to 625°C at 50°C/min; hold for 15 min. Heat at 30°C/min to 840°C; hold for 10 min. Cool at 30°C/min to 675°C; hold for 8 hr. Cool at 50°C/min to 600°C; hold for 10 hr. Cool at 50°C/min to 400°C; then at 2°C/min to room temperature. Total cycle time, approximately 22 hr.</p>
718-3	<p>Shorter version of standard cycle. The precipitation hardening soak at 600°C is shortened to 5 hr. Total cycle time, approximately 17 hr.</p>
718-4	<p>Precipitation harden, then seal and crystallize. Heat from room temperature to 675°C at 100°C/min; hold for 8 hr. Cool at 50°C/min to 600°C; hold for 10 hr. Cool at 50°C/min to 400°C, then at 2°C/min to room temperature. Heat to 1000°C at 100°C/min; hold for 8 min. Cool to 625°C at 50°C/min; hold for 15 min. Heat to 840°C at 30°C/min; hold for 10 min. Cool at 30°C/min to 650°C, at 5°C/min to 400°C; then at 2°C/min to room temperature. Total cycle time, approximately 22.5 hr.</p>
718-5	<p>Modified sealing and crystallizing cycle followed by "one step" hardening cycle. Heat from room temperature to 1000°C at 100°C/min; hold for 16 min. Cool to 650°C at 50°C/min; hold for 15 min. Heat to 800°C at 30°C/min; hold for 8 hr. Cool at 30°C/min to 650°C, at 5°C/min to 400°C; then at 2°C/min to room temperature. Total cycle time, approximately 13 hr.</p>
718-6	<p>Modified sealing and crystallizing cycle followed by "two step" abbreviated hardening cycle. Heat from room temperature to 1000°C at 100°C/min; hold for 16 min. Cool to 650°C at 50°C/min; hold for 15 min. Heat to 800°C at 30°C/min; hold for 20 min. Cool at 30°C/min to 700°C; hold for 2 hr. Cool at 30°C/min to 600°C; hold for 2 hr. Cool at 5°C/min to 400°C, then at 2°C/min to room temperature. Total cycle time, approximately 7 hr.</p>
625-1	As received.
625-2	Same as for 718-4.

Table 2 - RESULTS OF HARDNESS AND TENSILE TESTS

Batch No.	Hardness (Rockwell C)	Yield Strength (kpsi)	UTS (kpsi)
718-1	15	53.7 $\pm$ 3.0	114.9 $\pm$ 0.7
718-2	40	190.9 $\pm$ 6.7	195.8 $\pm$ 3.2
718-3	39	182.2 $\pm$ 7.5	197.0 $\pm$ 0.7
718-4	21	123.5 $\pm$ 8.0	140.0 $\pm$ 0.2
718-5	36	157.5 $\pm$ 1.8	193.2 $\pm$ 1.4
718-6	35	161.7 $\pm$ 3.0	193.2 $\pm$ 1.5
625-1	23	96.0 $\pm$ 1.6	152.3 $\pm$ 0.5
625-2	31	83.6 $\pm$ 0.6	145.7 $\pm$ 1.0

(a)

(b)

FIGURE 1 - Optical micrographs of Inconel 718 etched in HCl, 400X. (a) 718-1; (b) 718-6.

was already in a well-annealed condition. This observation is further illustrated by a comparison of the microstructures of the as-received and the heat-treated specimens (Figure 2). The heat-treated specimens exhibit some degree of secondary recrystallization, or anomalous grain growth, which can account for the observed decrease in strength.

The test data indicate that precipitation-hardened Inconel 718 is a much stronger material than Inconel 625, though a comparison of Figures 1 and 2 might suggest that the opposite is true. The Inconel 625 grains are much smaller than those of the Inconel 718; however, the effect of grain size on mechanical strength is

dominated by the effects of the hardening precipitates formed during heat treatment of the Inconel 718.

Summary

A modification to the standard heat treatment of Inconel 718 has been found that 1) leaves the material with a tensile yield strength greater than 150 kpsi and an ultimate tensile strength of more than 190 kpsi, and 2) is 15 hr shorter than the standard cycle. The considerable reduction of time spent at elevated temperatures minimizes the undesirable effects on a glass-ceramic material which is bonded to Inconel 718, permitting the fabrication of headers that possess good mechanical and hermetic integrity.

(a)

(b)

FIGURE 2 - Optical micrographs of Inconel 625 etched in HCl, 800X.
(a) 625-1; (b) 625-2.

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