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Y-1551

Engineering and Equipment

MASTER

MACHINING AND FINISHING FINE-GRAIN BERYLLIUM

V. M. Hovis

RELEASED FOR ANNOUNCEMENT
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UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the **ATOMIC ENERGY COMMISSION** under **U. S. GOVERNMENT Contract W-7405 eng 26**

**UNION
CARBIDE**

OAK RIDGE Y-12 PLANT
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V. M. Hovis

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ABSTRACT

The Oak Ridge Y-12 Plant has completed a study related to the machining and finishing of complex shapes made of fine-grained, high-oxide beryllium. Precision fabrication has been made possible by using a combination of conventional-machining and chemical-etching techniques. Work was done with both Grade S-200 and P-6 metal.

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SUMMARY

A capability has been established at the Y-12 Plant for machining and finishing complex structural components of high-strength, fine-grain beryllium. Precision fabrication has been made possible by using a combination of conventional-machining and chemical-etching techniques. Optimum strength properties in the components have been assured by the chemical-etching step which can be accomplished with predictable results. Through the use of consumable specimens and careful use of a nondestructive replica technique, a usable method for certifying the optimum strength conditions of beryllium components has been developed.

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INTRODUCTION

The Y-12 Plant has completed a study related to the machining and finishing of shapes made of fine-grained, high-oxide beryllium, designated Grade P-6 by the Brush Beryllium Company. The purpose of the study, jointly initiated by Sandia-Livermore and the Y-12 Plant, was to develop shop techniques and quality control methods that would assure optimum mechanical properties in completed hardware components which have been machined with care and precision.

Specific objectives of the program were to:

1. Establish machining procedures that would minimize surface damage in producing hardware items from Grade P-6 material.
2. Determine the depth of material which must be chemically etched from properly machined surfaces to remove twins and microcracks.
3. Develop an etching solution and technique with a reaction rate low enough to permit precise control of part dimensions within 0.5 mil during etching of the surface to the required depth.
4. Develop a nondestructive method for examining chemically etched surfaces of machined shapes for evidence of unremoved damaged material.
5. Conduct preliminary experiments on Grade S-200 beryllium and modify the results for Grade P-6 metal as required. This objective was necessary because Grade P-6 material was unavailable at the beginning of the program.

P-6 metal is significantly different from the classical hot-pressed grade. The average grain size is 7 to 15 microns and the beryllium oxide content is 5 to 7%. For comparison, Grade S-200 has an average grain size of 35 to 50 microns and a beryllium oxide content of 1.5 to 2%. In general, this material has a yield strength (0.2% offset) of 60,000 psi, an ultimate strength of 65,000 psi, and an elongation of 0.7% in a gage length of 1.0 inch at room temperature. These properties were determined from testing carefully prepared tensile specimens and are typical of the values inherent in the material.

The strength of beryllium, as determined from tensile specimens, is affected significantly by surface damage induced during the fabrication of the specimens. This damage is manifested in the form of microcracks at the surface and twinning of the surface layers. The damage is usually produced by the tools used in the machining process required to give shape and size to the specimens. Microcracks and twins provide areas for the multiplication of applied stresses during testing. The higher stresses at the roots of small cracks and at the edges of intercrystalline twins result in premature failure by the initiation of gross cracks that readily propagate through

the material. Under these conditions, the specimen exhibits a lower strength and ductility than the material inherently possesses. To determine more exactly the strength of beryllium and to reduce the scatter in the test data, the surface-damaged material of the tensile specimens is removed by a "stress-free" process. The most practical method is to chemically dissolve or etch the surfaces with a suitable reagent. For most beryllium, removal of material to a depth of 3 to 4 mils has been found to be sufficient to eliminate the effect of machining damage upon the strength. (1 - 3)

Whenever it is necessary to assure optimum strength in beryllium parts, equivalent to the strength of the tensile specimens, careful machining operations followed by chemical etching of the surfaces are required. For parts with close tolerances, thread forms, holes, and critical surfaces, it is found that the fabrication process must be controlled with particular attention to detail. In addition to dimensional quality control before and after etching, a practical nondestructive testing method is desirable for determining the effectiveness of the etching procedure in removing damaged material from the surface.

DISCUSSION OF THE STUDY

MACHINING

From an examination of various machined specimens, it appears that the depth of surface damage in the form of microcracks was about one mil in Grade S-200 material. Microcracks were difficult to produce to any significant depth in the P-6 material and those that did form extended less than one mil.

The depth of twinning within grains of S-200 material was greatly dependent upon the machining operations. A maximum depth of two mils was obtained from moderate grinding operations, whereas a depth of 3 to 20 mils was obtained from single-point tool action. The higher figure resulted from a heavy cut using a tool with an excessively worn cutting edge.

At the beginning of the program, very sharp cutting tools were used in all specimen preparation. A machining cut sequence was established consisting of two final roughing cuts at 10 mils followed by four finishing cuts at five, three, one, and one mils. Examination of the cutting tools revealed that a cut depth of one mil was impractical for the purposes intended. Tool wear, which occurred rather rapidly to a cutting edge radius of about 0.3 mil, destroyed the effectiveness of the one-mil cut depth. As a result, a final machining sequence was established for test machined shapes at ten, ten, five, and three mils with a maximum feed rate of three mils per revolution.

A number of tool materials have been tested on beryllium in the Y-12 Plant. It has been found that the best tool life is consistently provided by the straight tungsten carbide grades with minimum cobalt binder and with the smallest grain structure offered commercially. The addition of other alloying elements reduces tool life. Machining experience with P-6 material did not alter these conclusions.

DEPTH OF TWINNING WITH CONTROLLED MACHINING OPERATIONS

S-200 Material

Five groups of four each tensile bars, as shown in Figure 1, and five groups of four each tensile rings, as shown in Figure 2, were machined from Grade S-200 beryllium stock. For both the bars and rings, one group was etched to remove one mil of material from all surfaces, one group to remove two mils, one group four mils, one group eight mils, and one group ten mils. All specimens were machined under identical conditions with a finishing sequence of a five, three, one, and one-mil cut depth using single-point tools. The critical dimensions of the specimens were measured with a high degree of precision before and after etching.

Notes:

- (1) Decimal tolerance is ± 0.001 ", and fractional tolerance is $\pm 1/64$ ".
- (2) Finish cut depths conformed to the following sequence: five, three, one, and one mil as measured on the radius.
- (3) The speed was 450 rpm and the feed was three mils per revolution.
- (4) Gage length was tapered, each end being slightly larger than the midpoint.

Figure 1. DIMENSIONAL REQUIREMENTS FOR THE TENSILE BAR USED IN THE GRADE S-200 BERYLLIUM TESTS.

Notes:

- (1) Tolerance was one mil.
- (2) The final four machining cuts were five, three, one, and one mil in depth as measured on the radius or face of each surface and they were in the sequence listed.

Figure 2. DIMENSIONAL REQUIREMENTS FOR THE TENSILE RING MADE FROM S-200 BERYLLIUM.

The specimens were examined by use of a binocular light microscope for evidence of surface damage induced during machining. Microcracks, smeared material, and laminations observed on the machined surfaces appeared to be completely removed by etching to a depth of one mil. Below this depth, damaged material was evidenced by the presence of twinned grains or crystals. Therefore, particular attention was given to the relative numbers of twinned crystals observed on the specimen surfaces as a measure of the amount of machine-induced damaged material remaining after etching.

Using polarized light, twinned crystals were readily observed on etched surfaces directly and on surface replicas taken from the specimen surfaces under study. Using acetate replicating tape, softened slightly in acetone and pressed hard to the specimen area under investigation, satisfactory detail was obtained to permit good identification of the typical configuration of twins.

Examination of the specimens was accomplished by counting the number of twinned crystals observed in 20 randomly selected microscope fields at 400X. The total number of twinned crystals was recorded for each set of 20 fields. The following surfaces were checked:

Tensile Rings - End surfaces, 10 fields per surface by direct examination.

Tensile Rings - End surfaces, 10 fields per surface by replica examination.

Tensile Rings - Outside surface, 20 fields per surface by direct examination.

Tensile Rings - Outside surface, 20 fields per surface by replica examination.

Tensile Rings - Inside surface, 20 fields per surface by replica examination.

Tensile Bars - Gage length surface, 20 fields per surface by direct examination.

Tensile Bars - Gage length surface, 20 fields per surface by replica examination.

The data, obtained from the study of S-200 grade beryllium only, are presented in Figure 3. Apparently the damaged material induced by controlled machining operations was removed by removal of the material to a depth of 3.5 to 4.5 mils. It appears that a small number of twinned crystals, produced by the material preparation process, exists throughout the material.

P-6 Material

To gather data relative to the problem of surface quality certification, a study was made of the effect of surface condition upon the mechanical properties of P-6 grade material. The solid end of an experimental P-6 pressing was cut into pieces for tensile specimens. The pieces were shuffled to randomize their positions within the block and then divided into four groups of ten each. Each group was machined as shown in Figure 1 for Grade S-200 material except for the sequence of finishing cuts which was

10, 10, 5, and 3 mils as measured on the radius and inspected. Conventional shop-ground cutting tools were used. One group was not etched, a second group was etched to remove one mil from all surfaces, the third group had two mils removed, and the fourth group eight mils. All etched specimens were reinspected.

Figure 3. DATA OBTAINED FROM TWENTY TENSILE BARS AND TWENTY TENSILE RINGS MADE FROM S-200 MATERIAL.

The specimens had been identified for shop purposes by an orderly numerical sequence for each group of ten. To provide a statistically balanced set of test data, the 40 specimens were pulled in a statistically determined random sequence. The test data were statistically analyzed and the following results were obtained:

1. Etching 0, 1, 2, and 8 mils did not affect the yield strength.

2. The ultimate strength and the percent elongation for the 0-mil etch were significantly lower than the results for the 1, 2, and 8-mil etches. The results for the 1, 2, and 8-mil etches were not significantly different.

The means, differences, and limits of error (95% confidence) for the various tests are summarized in Table 1. A summary of the test data for the forty P-6 beryllium specimens is presented in Table 2.

Table 1
TENSILE BAR TESTS ON GRADE P-6 MATERIAL

Test	Etch (mils)	Mean of Ten Specimens ($\pm$ LE of the mean)	Difference Between Lowest and Next Lowest ($\pm$ LE of the difference)
Yield Strength at 0.2% Offset (psi)	0	60,075 $\pm$ 1150	775 $\pm$ 1620
	1	59,255 $\pm$ 1150	
	2	60,030 $\pm$ 1150	
	8	60,500 $\pm$ 1090 ⁽¹⁾	
Ultimate Tensile (psi)	0	63,888 $\pm$ 1060 ⁽²⁾	2284 $\pm$ 1460 ⁽⁴⁾
	1	66,433 $\pm$ 1000 ⁽³⁾	
	2	66,172 $\pm$ 1000 ⁽³⁾	
	8	66,312 $\pm$ 1060 ^{(1) (3)}	
Elongation (%)	0	0.575 $\pm$ 0.081 ⁽²⁾	0.133 $\pm$ 0.111 ⁽⁴⁾
	1	0.796 $\pm$ 0.076 ⁽³⁾	
	2	0.708 $\pm$ 0.076 ⁽³⁾	
	8	0.774 $\pm$ 0.081 ^{(1) (3)}	

- (1) Only nine results available; one specimen broke before yielding.
 (2) Two specimens broke at the cap and results omitted.
 (3) One specimen broke at the cap and result omitted.
 (4) Significantly different.

Preliminary experience with typical P-6 material received from Brush Beryllium Company demonstrated that single-point tool machining does not produce cracking and twinning of crystals in this metal to the depth observed in S-200 grade material. This observation appears to be confirmed by the work on the 40 tensile specimens. The difference in the depth of the machining damage is attributed to the difference in the average grain size and the beryllium oxide content. S-200 material, with an average grain size of 30 to 40 microns (approximately 1.4 mils), appeared to propagate surface deformation and twinning produced by a cutting tool to a depth equivalent to two grain layers or three mils. Beyond this depth, no significant effect was seen. Also,

Table 2
TENSILE BAR TESTS ON FORTY P-6 BERYLLIUM SPECIMENS

Specimen Number	Yield Strength ⁽¹⁾ (psi)	Ultimate Strength (psi)	Elongation ⁽²⁾ (%)
1	56,050	62,250	0.72
2	60,650	64,200	0.52
3	61,050	64,400	0.52
4	64,450	63,500	0.48
5	61,600	66,250	0.66
6	60,350	64,050	0.56
7	61,250	64,700	0.52 ⁽³⁾
8	56,250	60,900	0.58
9	60,550	63,550	0.48 ⁽³⁾
10	61,550	65,550	0.56
11	59,650	68,150	0.96
12	57,300	65,250	0.82
13	61,050	67,700	0.72
14	60,750	67,800	0.80
15	57,700	64,850	0.78 ⁽³⁾
16	61,000	64,400	0.57
17	61,050	66,100	0.69
18	57,300	64,300	0.80
19	56,650	67,100	1.00
20	60,100	67,100	0.80
21	60,500	67,000	0.74
22	56,400	63,600	0.76
23	61,000	66,850	0.74 ⁽³⁾
24	60,900	65,450	0.59
25	60,900	65,450	0.60
26	61,300	65,200	0.60
27	61,000	66,400	0.74
28	57,600	67,150	0.70
29	59,700	67,600	0.88
30	61,000	67,700	0.76
31	58,850	67,450	1.01
32	60,350	63,300	0.56
33	60,350	67,400	0.80
34	(4)	(4)	(4)
35	60,700	66,300	0.72
36	61,950	64,250	0.49 ⁽³⁾
37	61,250	66,950	0.72
38	61,150	66,000	0.71
39	59,850	66,500	0.83
40	60,050	66,600	0.84

(1) 0.2% offset.

(2) In one-inch gage length.

(3) Broke at cap.

(4) Broke before yielding.

microcracks were not observed to extend in depth beyond one mil. If the same general mechanism of twinning applies to P-6 material with an average grain size of 10 to 15 microns (approximately 0.5 mil), then the propagation of twinning effects should not extend much beyond one mil. Also, because the ductility of P-6 material was equal to that of Grade S-200, microcracks produced by machining should not extend beyond the depth of one mil.

Another study was made to learn more about the depth of surface damage produced in P-6 beryllium by cutting tools. Six samples of P-6 material (1" D x 1/2" L) were cut from the same pressing obtained from Brush Beryllium Company and were used for the tensile bar work. The specimens were prepared according to the following procedure:

1. The six P-6 specimens were stamped on one face with the designations: P6-L, P6-0, P6-1, P6-2, P6-3, and P6-4.
2. The other face was machined with a final cut of 10 mils in depth at a feed rate of 10 mils per revolution using a standard commercially ground cutting tool. A tool with an ultrasharp cutting edge was not used.
3. Specimen P6-L was lapped on the machined surface using the same methods that were used to hand polish hardware components.
4. All specimens were examined microscopically and photographic records were made of the representative surface conditions observed for each specimen.
5. Specimens P6-L and P6-0 were etch polished on the machined face using Linde "A" polishing grit and a dilute oxalic acid vehicle. Material removed was not less than 300 and not more than 500 microinches in depth.
6. Specimens P6-1 through P6-4 were chemically etched using the 10% nitric-90 % phosphoric acid solution developed for beryllium etching. The amounts removed from the surfaces of each specimen were: 1 mil from P6-1, 2 mils from P6-2, 3 mils from P6-3, and 4 mils from P6-4.
7. Specimens P6-1 through P6-4 were etch polished on the machined and chemically milled face. The procedure was the same as for Specimens P6-L and P6-0. Material removed was not less than 300 and not more than 500 microinches in depth.
8. All specimens were reexamined by microscope for evidence of residual surface-damaged material. Photographic records were made of the representative surface conditions observed for each specimen.

The etch-polish procedure in conjunction with polarized light was necessary to bring out the twinned areas in P-6 material when examined by the microscope. Chemical milling alone was not sufficient.

After etch polishing, all specimen surfaces were carefully examined by microscope using both phase-contrast and polarized light. The results are summarized in the paragraphs that follow.

P6-0 - Surface smearing by the cutting tool was still very evident. Twinning was very hard to detect because of the surface condition, and microcracks were difficult to identify in the view presented. Figure 4 shows a typical surface area.

P6-L - The surface was relatively smooth because the machining marks had been lapped out by shop personnel using techniques applicable to hand polishing of parts during fabrication. Twinned crystals were very hard to find and microcracks were not observed. Figure 5 shows a typical surface area.

P6-1 - The grain structure was fairly well defined. A fine-grain, high-oxide structure was well presented. Very few twins were found over the entire surface. When found, they were located in large grains—50 microns or larger. Microcracks were not observed. A typical surface area is presented in Figure 6.

P6-2 - The grain structure was well defined. A search of the entire surface revealed very few twinned grains. When found, they were in large crystals—50 microns or larger. Microcracks were not observed. Figure 7 is a photograph of a typical surface area.

P6-3 - The grain structure was well defined. There were more twins in this specimen surface than in any of the others. A detailed study revealed a much larger proportion of large grains to small ones when compared with the other specimens. In the grains smaller than 30 to 40 microns in diameter, no twins were found. In the grains 50 microns in diameter or larger, twinning was found more readily. In grains 120 to 150 microns in diameter, finding twinning was almost a certainty. This specimen surface appeared to be more typical of S-200 material in many spots than P-6 material. No microcracks were observed. Figure 8 shows a typical twinned area; Figures 9 and 10 are photographs of a different surface area showing the comparison between the use of polarized and phase-contrast illumination.

P6-4 - The grain structure was well defined and was typical of fine-grain, high-oxide P-6 material. A careful search over the entire specimen area did not reveal any clearly defined twins. Microcracks were not observed. Figure 11 shows a typical surface area.

The information obtained from the study of the 40 tensile specimens and the study of the six discs is significant. From an analysis of these data, it appears that P-6 beryllium is sufficiently different from other types to justify a reduction in the chemical milling requirements. The findings seem to support the conclusion that chemical removal of material to a depth of 1 to 2 mils in P-6 structural shapes is sufficient to assure optimum mechanical properties. With a predominate grain size no larger than 15 microns and with a beryllium oxide content of about 5%, the depth of surface damage produced by good machining techniques cannot be expected significantly to exceed one mil.

Figure 4. POLISHED SURFACE OF SPECIMEN P6-0. (Polarized Light; 1500X)

Figure 5. POLISHED SURFACE OF SPECIMEN P6-L. (Polarized Light; 1500X)

Figure 6. POLISHED SURFACE OF SPECIMEN P6-1. (Polarized Light; 1500X)

Figure 7. POLISHED SURFACE OF SPECIMEN P6-2. (Polarized Light; 1500X)

Figure 8. SURFACE OF SPECIMEN P6-3 SHOWING TYPICAL TWINNED AREA. (Polarized Light; 1500X)

Figure 9. VIEW OF ANOTHER PORTION OF THE SURFACE OF SPECIMEN P6-3. (Using Polarized Light at 1500X)

Figure 10. SAME VIEW AS THAT SHOWN IN FIGURE 9. (Using Contrast Illumination at 1750X)

Figure 11. POLISHED SURFACE OF SPECIMEN P6-4. (Polarized Light; 1500X)

It is apparent that a chemical etching procedure sufficient to remove microcracks and smooth out the microscopic rough surfaces produced by cutting tools will be adequate for the removal of twinned crystals and dislocations in good P-6 grade beryllium.

When compared with the four-mil etching depth required of S-200 grade material, the reduced etch depth of P-6 material offered several advantages: (1) there was an improvement in the precision with which completed parts were furnished as etched; and (2) the etching time was reduced by about 50% which also improved the uniformity of the surface conditions and dimensions of finished parts by reducing the effects of gas-bubble scouring and differential-agitation velocities.

CHEMICAL ETCHING

Particular attention was devoted to the etching solution. It was desirable to have a solution that would: (1) provide a smooth, reflective surface capable of observation by light optics; (2) tend to accentuate twinned structures in crystals; and (3) remove material slowly enough so dimensional control could be facilitated during the final chemical-milling process. Laboratory experiments with small cylinders of S-200 hot-pressed beryllium and a variety of reagents demonstrated that a solution of 10 vol % HNO_3 (70%) + 90 vol % H_3PO_4 (85.6%) at 75°C would give good results. After conditioning this etchant at 75°C for 16 hours, bright surfaces were produced that readily revealed twinned crystal structures when viewed with an optical microscope at 200 to 400X. The material removal rate was from 4 to 6 mils per hour, an improvement over the rate of 2 to 3 mils per 5 minutes commonly encountered with other etchants.

During the chemical etching process, small gas bubbles continuously evolved and swept upward around the part surfaces as they escaped from the bath. This streaming of the gas bubbles produced an erosion effect around holes and raised surfaces that marred the surface quality. This effect was especially noticable when attempts were made to remove four mils from the surfaces of the larger test shapes. The effect was most pronounced around the discontinuities in the surfaces paralld to or slightly inclined to the upward direction of the bubble streams. Figure 12 shows a schematic representation of the effects observed with typical erosion length.

By the use of test shapes made of S-200 grade beryllium, an agitation process was developed that minimized the gas-bubble effect. Figure 13 shows a typical shape used for study. It was necessary to provide agitation around a vertical axis. This action was achieved by oscillating the part. The rotary motion and frequency was very nearly like that used by the agitator system in a domestic washing machine. Tests were conducted on various large shapes designed to simulate thread problems and dimensions that might occur in fabricating actual hardware shapes. Figures 14 and 15 show typical test pieces made of S-200 grade material.

The etching and agitation techniques developed on S-200 material were repeated on P-6 material. No changes were required to produce the required results.

Figure 12. GAS-BUBBLE SCOURING EFFECT.

REPLICATION

To meet the requirement for a nondestructive method of examining etched surfaces for evidence of residual surface damage, the best practical technique for development appeared to be that of surface replication. Work with the solvent films that give the best detail for electron microscopy was discouraging. These films adhered so tightly to the metal that they had to be broken loose with a sharp-edged tool before stripping from the surface. This procedure resulted in harmful scratches in the metal surface at the film edges.

Good results were obtained with the conventional cellulose acetate films. With film pieces 5 to 10 mils in thickness, softened on the replicating side with an acetone-cellulose acetate solution, and pressed to the surface until dry, adequate reproductions of surface detail were obtained on S-200 material. Figure 16 is a photograph of a machined surface of S-200 beryllium, chemically milled to remove approximately one mil of material, as viewed directly by the microscope at 400X. A number of twinned crystals can be readily observed. Figure 17 is a photograph of a replica surface taken from the same specimen surface described for Figure 16, as viewed by the microscope at 400X. This replica surface, which was unshadowed, clearly presents a large number of twinned crystals that were present in the original surface.

Figure 14. ETCHING TEST PART 2.

Figure 15. ETCHING TEST PART 3.

R-001-4(U)

Figure 16. SURFACE OF MACHINED S-200 BERYLLIUM TENSILE RING CHEMICALLY MILLED ONE MIL.
(~400X)

R-001-4(U)

Figure 17. SURFACE REPLICA OF MACHINED S-200 BERYLLIUM TENSILE RING CHEMICALLY MILLED ONE MIL. ($\sim 400\times$)

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Figure 18. SURFACE OF MACHINED S-200 BERYLLIUM TENSILE RING CHEMICALLY MILLED FOUR MILS. (~400X)

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Figure 19. SURFACE REPLICA OF MACHINED S-200 BERYLLIUM TENSILE RING CHEMICALLY MILLED FOUR MILS. ($\sim 400\times$)

ground cutting tools) and then etch polished to remove 300 to 500 microinches before a significant twin population could be revealed. As previously described and as shown in Figures 4 through 11, the surface damage from light machining cuts did not extend to any significant depth in P-6 material having an average grain size of 15 microns. It was learned from the material represented in Figures 8 through 10 that in localized areas within P-6 pressings it is possible to have a number of large grains in the size range of 50 to 100 microns. Where such areas occurred, the depth of surface damage from machining was about 3 mils. Such a condition may not affect the strength of hardware parts etched to a depth of 1 to 2 mils if an area of noncritical loading is affected.

The simple replica technique that was so useful on S-200 material did not seem to be practical when applied to the P-6 grade beryllium. The surface condition produced by an etchant tended to diffuse light to such an extent that a clear definition of grain detail was prevented in optical microscope examination. It appeared that the diffused surface was produced by a rounding of the grain surfaces produced by a preferential etching action at the grain boundaries. The effect was less pronounced on S-200 material because the grains were larger and the beryllium oxide grain boundaries were smaller in width.

Application of metallographic laboratory techniques was required to bring out the structure; and, at the moment, this does not seem to be practical when applied to complex shapes. Also, the apparent lack of surface damage propagation in P-6 material to a significant depth during machining cast doubt upon the need for surface quality certification after a proper chemical milling treatment.

For purposes of checking critical areas of P-6 parts for grain structure integrity, the cellulose acetate replica has value. In spots or bands where large grains existed (30 microns in diameter or larger), the action of the nitric-phosphoric etching solution was very much like the action on S-200 grade beryllium. The grain structure was presented by the etchant and twins were revealed. Application of the cellulose acetate replica to such an area reproduced the structure with sufficient fidelity for identification by the microscope. If the average grain size in a critical area was small and representative of P-6 material, the application of replicas to sample the surface after etching would probably not reveal any significant data.

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CONCLUSIONS

From this study, the following conclusions have been reached:

1. Where single-point cutting tools were used either individually as in a lathe operation or as multiple units in a milling cutter or rotary form cutter, it was found that the final four cuts should be 10, 10, 5, and 3 mils in depth at a maximum feed rate of 3 mils per revolution. For most machining operations, cut depths less than 3 mils were found to be impractical. Tool wear readily produced a rounding of the cutting edge which destroyed the effectiveness of the smaller cut depths of 1 to 2 mils.
2. Cutting-tool material should be a fine-grained tungsten carbide with a maximum cobalt binder content of 6%. The addition of other alloying elements tended to reduce wear resistance of the cutting edge.
3. For S-200 grade beryllium, surface removal to a depth of 4 to 5 mils by chemical etching was necessary to remove twins and microcracks; for P-6 grade beryllium, etching to a depth of 1 to 2 mils removed the surface damage from properly machined material.
4. Material removal rate with a chemical etching solution consisting of 10 vol % concentrated nitric acid (70%) and 90 vol % concentrated phosphoric acid (85%) at a temperature of 70° C was about 4 mils per hour. Bright, reflective surfaces were produced after the solution had been conditioned by heating at 75° C for 16 hours.
5. Machined shapes immersed in the etching solution required agitation around a vertical axis with an oscillating rotary motion (washing machine agitator). This action was necessary to minimize a scouring effect upon part surfaces produced by gas bubbles streaming around discontinuities such as holes and pads.
6. It was found by direct optical examination of machined and etched surfaces of S-200 material that the nitric-phosphoric solution revealed clearly the twinned crystals and microcracks still present after etching. The etching solution tended to remove material at a slightly higher rate at the grain boundaries and thus rounded the grain edges. A significant flat portion of the grain surfaces remained for examination.
7. Direct optical examination of the surface of machined and etched P-6 material did not reveal twins and microcracks. Because of the smaller grain size and the edge-rounding effect, the surfaces of the grains were rounded to such an extent that the reflected light was diffused.
8. For an examination of P-6 material after etching, a method of etch polishing was

developed that would clearly show the surface condition. The method used consisted of polishing with Linde "A" polishing grit with a 10% oxalic acid-distilled water vehicle to remove from 300 to 500 microinches of material.

9. A method of replicating the surface of etched S-200 material was developed using conventional cellulose acetate tape. The tape was softened in acetone, pressed to the surface until dry, removed, and examined directly without shadowing to accentuate the details. Replica quality was sufficient to clearly reveal the presence of twins and microcracks. With this technique, representative surface areas of completed hardware items can be checked for the presence of residual damaged material.
10. The cellulose acetate replica technique was not applicable to machined and etched P-6 material. The grains were too small for accurate reproduction of the detail required. It was found, however, that in areas where the grain sizes may inadvertently exceed 35 microns in size, the replica method will clearly reproduce the surface condition.

REFERENCES

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