JOURNAL OF MATERIALS SCIENCE 8 (1973) 1788–1794

The effect of surface finish on the pressure-induced ductility of beryllium

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An investigation has been made of the effect of specimen surface finish and sheathing on the tensile properties of both cast and extruded ingot and hot-pressed powder beryllium, under the influence of an applied hydrostatic pressure of 78.7 kg mm^{-2} (7.72 kbar). Three surface finishes – as-machined, chemically etched and electropolished – were investigated. For each surface finish, specimens of both materials were tested with their gauge length sections bare and sheathed with a rubberized coating. The chemical etching and the electropolishing treatments were designed to remove the damage induced by the specimen machining operations and the rubberized coating was applied to prevent the pressurizing fluid wetting the specimens. Other workers have claimed that electropolishing alone is sufficient to enable the maximum ductility of beryllium to be realized in a hydrostatic environment. However, the present investigation shows that both a post-machining surface treatment and the application of a rubberized coating are necessary before this condition is attained. The data also suggest that beryllium exhibits a Rebinder effect.

1. Introduction

Beryllium is susceptible to machining damage which has a deleterious effect upon the mechanical properties of the metal. Consequently, at normal ambient temperature, a damaged tensile test piece shows a marked reduction in both its ultimate tensile strength and ductility, as compared with an undamaged specimen. Machining operations can give rise to deformation twinning [1-3], microcrack formation [1, 2] and internal stress [1, 3]. The relative importance of these effects in the nucleation and/or propagation of brittle cracks has not been unequivocally established. Nevertheless, it has been qualitatively shown that machining damage can be alleviated by annealing machined parts at a temperature sufficient to recrystallize the twinned material and/or etching the damaged surface to a depth of 0.12 to 0.25 mm [2, 4].

The effect of an applied hydrostatic pressure on the tensile properties of beryllium has been investigated by a number of workers [5-11]. Extruded ingot [6, 7, 10], rolled ingot [11], hot-pressed powder [6, 8-10] and hot-pressed and rolled powder sheet [6, 9] materials have been studied in these investigations. Generally, the machining damage induced in the test pieces was removed by either chemical etching [6, 9], chemical etching and annealing [7, 8] or electropolishing [10, 11]. However, in some instances where comparable experimental conditions and materials and ostensibly damage-free specimens were used, the results of the different investigators show a wide divergence.

Bridgman [5] made a limited investigation of the tensile behaviour of a number of brittle materials under the influence of an applied hydrostatic pressure. He tested samples both with and without their gauge sections sheathed in copper; the sheathing being intended to prevent the specimens being wetted by the pressurizing fluid. However, he was not able to show that sheathing beryllium had any beneficial effect upon its properties although it markedly improved those of a variety of cast iron which was completely brittle in the absence of a sheath. Aladag *et al* [10] claimed that sheathing beryllium with rubber produced no improvement in ductility over that obtained by removing the machining damage by careful electropolishing.

In complete contrast, Stack and Bobrowsky [6] reported that the use of a rubber-like sheath enhanced the ductility of chemically etched beryllium samples as compared with that obtained for similar specimens which had been exposed to the pressurizing fluid during testing. Bridgman [5] did not specify the nature of the beryllium which he used in his experiments. However, the results of the other investigators [6, 10], were applicable to both the extruded ingot and hot-pressed powder grades of beryllium.

It can be seen from the above account of the previous work that there is a considerable conflict of evidence concerning the effect of surface finish on the pressure induced ductility of beryllium. The major variables that have been involved in the previous investigations include the type of beryllium, the nature of the post-machining treatments and the use of a sheath to prevent wetting by the pressurizing fluid. However, it has not been found possible to rationalize all the experimental data. Consequently, it was decided to make a detailed investigation of the effect of surface finish on the pressure-induced ductility of both hot-pressed powder and extruded ingot beryllium. In addition, it was considered that such an investigation was a necessary preliminary to investigating the pressure dependence of the ductility of the same two grades of beryllium.

TABLE I Analytical data for the experimental materials

	O	C	Fe	Al	Si
Ingot material	100	470	180	30	150
Powder material	8700	1070	1470	205	420

ppm by weight

2. Experimental materials and methods

2.1. Materials

The compositions of both the extruded ingot and hot-pressed powder grades of beryllium used in the investigation are given in Table I. The extruded bar was produced by sheathing a cast ingot in a mild steel can and subsequently extruding the composite billet at 1050° C, through a 40:1 reduction ratio. The resulting bar was acid desheathed in concentrated nitric acid to leave a beryllium bar approximately 1.25 cm diameter. The hot-pressed block was produced by vacuum hot-pressing −250 mesh beryllium powder in a graphite die. A load of approximately 0.8 kg mm^{-2} was applied at 600° C and maintained while heating to 1100°C, and for a period of 1 h thereafter. The load was then removed and the hot-pressing allowed to cool freely to room temperature. After fabrication, the grain sizes of the hot-pressed and ingot grades of beryllium were 15 and 250 μm respectively.

Conventional screw ended tensile test pieces, with a gauge length of 1.015 cm and a gauge diameter of 0.4 cm were turned from both materials using a series of diminishing cuts, with a final cut of 0.0125 mm. This machining procedure was designed to minimize the amount of damage induced into the specimens by the turning operation.

2.2. Methods

2.2.1. Chemical etching and electropolishing

The depth to which metallographically observable machining damage affects beryllium is grain-size dependent; the depth of the damaged layer increasing with increasing grain size. Therefore, in order to produce damage-free specimens, the gauge length surfaces had to be etched or polished to a depth characteristic of the material grain size. Metallographic examination showed that this condition was achieved by removing approximately 0.25 mm/surface from the ingot specimens and 0.1 mm/surface from the hot-pressed powder samples.

The surface layers were removed using both chemical etching and electropolishing. In the former case, a 10% H_2SO_4 solution was used at ambient temperature. The electropolishing was carried out with a p.d. of 20 V across a cell consisting of a specimen and a stainless steel cathode immersed in an electrolyte composed of 3 vol H_3PO_4, 1 vol H_2SO_4, 1 vol glycerine and 1 vol ethyl alcohol. During the polishing operation, external cooling of the cell was necessary in order to maintain the electrolyte temperature at its normal ambient value.

To enable the evaluation of the effect of surface finish to be carried out, four specimens of each material were left in the as-machined condition, four were chemically etched and four were electro polished. Subsequently, two specimens from each group were sheathed in rubber by dipping them in a mixture of a proprietary rubber based adhesive and ethyl methyl ketone. This gave an average sheath thickness of 0.125 mm, which was intended to prevent the wetting

of the specimen surfaces by the pressurizing fluid.

2.2.2. Metallography and scanning electron microscopy

The effect of the machining and the subsequent post-machining treatments on both the structure and surface finish of the specimens was investigated using optical metallography and scanning electron microscopy. Bar samples for the microscopy were turned in a manner identical to that used for the gauge length sections of the tensile test pieces. The surfaces of these samples in the as-machined, chemically etched and electropolished conditions, were examined using scanning electron microscopy at a potential of 20 kV. Prior to examination, the specimens were ultrasonically cleaned and then mounted in the microscope chamber so that they were inclined at an angle of 45° to the electron beam. Subsequently, the as-machined and chemically etched samples were metallographically examined. These specimens were mounted in plastic and their end faces, that is, the faces normal to the machined surfaces, were prepared using conventional hand grinding and diamond polishing techniques. The final polishing was done on a selvyt cloth impregnated with an aqueous suspension of gamma alumina. During the course of the final polishing operation, the specimens were periodically etched in a 2% hydrofluoric acid solution. The microscopic examination was carried out using polarized light.

2.2.3. Mechanical testing

The tests were carried out in a high pressure tensile rig which has previously been described in detail [12]. A mixture of castor oil and methanol was used as the pressurizing medium and all the tests were carried out at a hydrostatic pressure of 78.7 kg mm^{-2} (7.72 kbar). The pressure was generated by forcing the top plunger into the container and thereby compressing the fluid. The specimens were subjected to a tensile load by lowering the bottom ram of the press which supported the bottom plunger. The specimen load was measured by an internal load cell, the movement of the bottom plunger, which is an indication of the specimen extension, by a linear potentiometric stroke gauge and the fluid pressure by a manganin pressure gauge. These measurements were recorded on a multi-channel strip chart recorder. It was not possible to control the strain-rate of the tests very precisely, however, the majority of the tests were carried out using a strain-rate in the range 0.005 to 0.007 sec^{-1}.

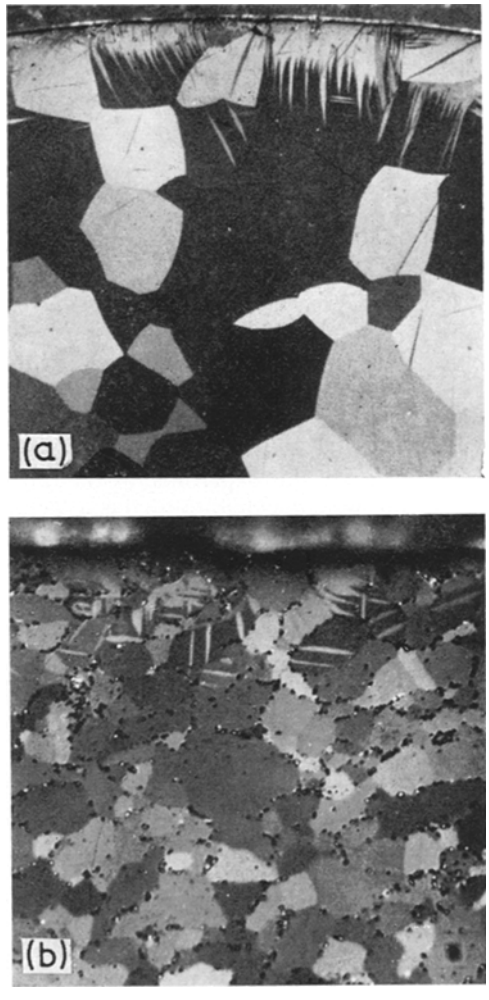


Figure 1 The damage induced by the specimen machining operation. (a) Extruded ingot beryllium (× 60); (b) hot-pressed powder Beryllium (× 385).

3. Experimental results

3.1. Optical and electron metallography

Fig. 1 shows the structure of the two grades of beryllium investigated, in the as-machined condition. As can be seen, the metallographically observable machining damage consists of a peripheral layer of heavily twinned grains. However, in the case of the ingot material some microcracking is also apparent. Fig. 2 shows

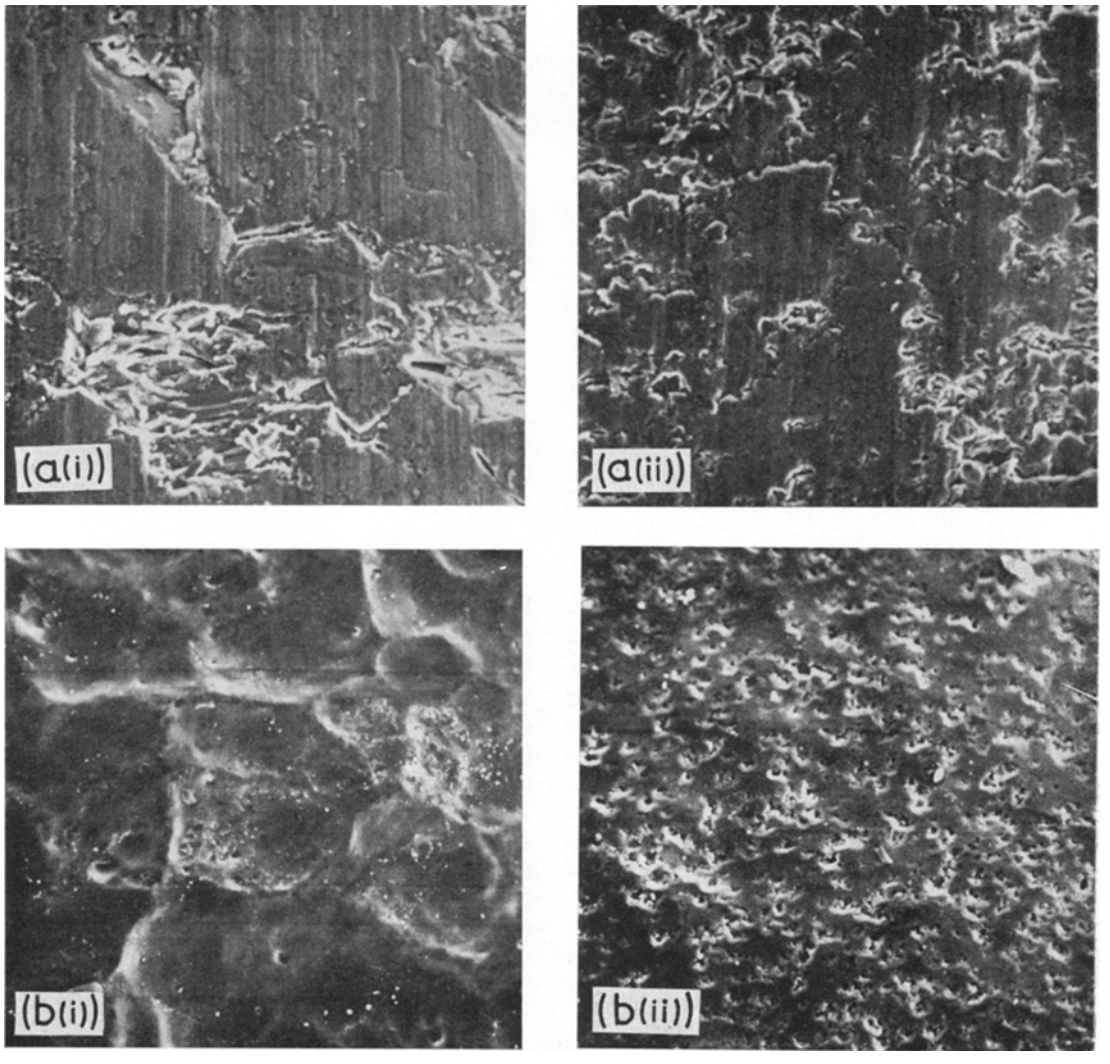
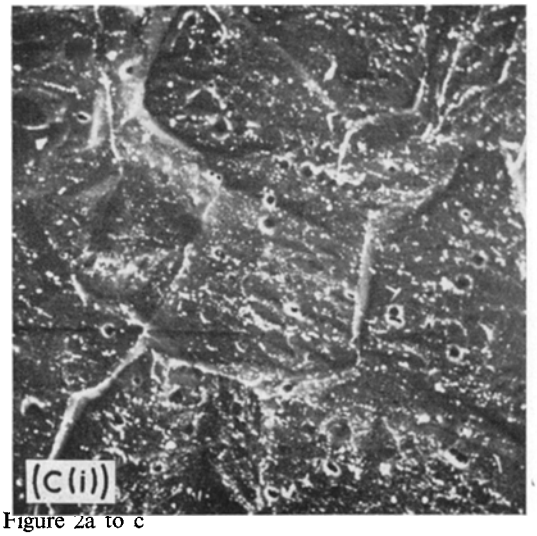


Figure 2 Scanning electron microtopographs of the surfaces of the ingot and hot-pressed powder samples in the as-machined condition and after post-machining treatments (× 115). (a) As-machined, (i) ingot, (ii) hot-pressed powder. (b) As-machined and electropolished, (i) ingot, (ii) hot-pressed powder. (c) As-machined and chemically etched, (i) ingot, (ii) hot-pressed powder.

scanning electron microtopographs of both the ingot and hot-pressed powder specimen surfaces in the as-machined, chemically etched and electropolished conditions. The as-machined samples clearly show the parallel markings left by the lathe turning operation. In addition, some cracks are visible in the ingot sample, which because of their straightness are presumed to be basal cleavage cracks. Irregular fissures can be seen in both samples and these are thought to arise as a result of inclusion tear-out and of grain cleavage. The microtopographs show that both the electropolishing and chemical etching post-machining treatments are effective in removing the cracks and fissures. However, the electro-polishing has clearly delineated the grain structure of the ingot material and the sites of inclusions in the hot pressed powder sample. In contrast, the chemical etching has revealed the grain structure as well as the inclusion sites of both materials. In the case of the powder sample however, the inclusions tend to be obscured by the grain boundaries present in the structure.



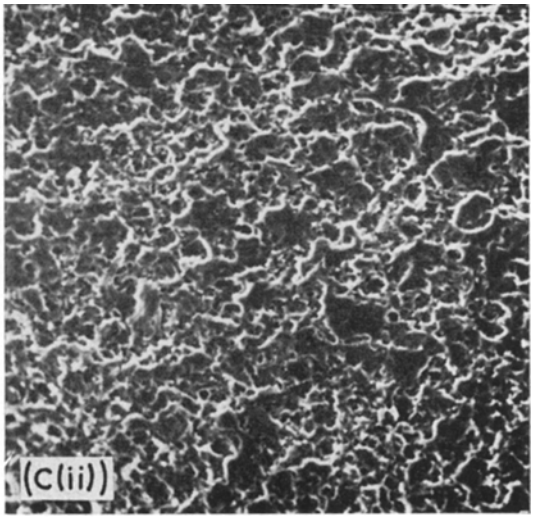


Figure 2a to c

3.2. Mechanical testing

Table II shows the tensile test data obtained for the two grades of beryllium examined. Generally, the results represent the mean of two separate tests, however, in three cases as indicated in the table, only one satisfactory test result was obtained. Unfortunately, because of the limited amount of experimental material available, it was not possible to produce additional specimens to enable duplicate tests to be run in these instances.

Quantitatively, in the case of the ingot material, the repeatability of the ductility data was better than 15% and of the strength data, about 6%. The hot-pressed beryllium results showed that the disparity between repeat ductility results was less than 15% and in the case of the strength data about 7%. None of the bare hot-pressed powder samples reached the point of instability. Consequently, no UTS values are given in Table II for these specimens. Instead nominal fracture stress values are quoted, where the nominal fracture stress is defined as the (fracture load/original cross sectional area). By analogy, it is thought that the result given in Table III as a UTS value for the hot-pressed powder material

TABLE II The effect of surface finish on the tensile properties of beryllium

	Surface condition		% Elongation	% R of A	UTS (kg mm^{-2})	Nominal fracture stress (kg mm^{-2})	True fracture stress (kg mm^{-2})
Ingot material	As-machined	Bare	36.1	36.1	50.9		62.6
		Sheathed	42.8	44.9	50.9		78.6
	Chemically	Bare*	47.4	41.3	48.5		71.2
	etched	Sheathed	48.0	54.5	50.2		87.2
	Electropolished	Bare	44.2	48.4	46.6		76.4
		Sheathed	47.8	56.0	51.8		88.4
Powder material	As-machined	Bare*	10.0	8.7		44.3	48.6
		Sheathed	47.6	36.5	61.3		98.6
	Chemically	Bare	14.1	12.3		53.4	60.9
	etched	Sheathed	55.6	53.6	60.8		12.0
	Electropolished	Bare*	17.8	13.8		55.4	63.7
		Sheathed	61.5	54.3	60.5		112.4

*One result only.

TABLE III Published data giving the mechanical properties of unsheathed extruded ingot and hot-pressed powder beryllium specimens tested under a hydrostatic pressure of 78.7 kg mm^{-2}

	Ref.	Surface condition	% Elongation	% R of A	UTS (kg mm^{-2})	True fracture stress (kg mm^{-2})
Ingot material	[6]	Chemically etched to remove 0.15 mm/surface	18	26	45.5	
	[7]	Chemically etched to remove 0.1 mm/surface	29	29		
	[10]	Electropolished to remove 0.05 mm/surface		56.5		91.6
Powder material	[6]	Chemically etched to remove 0.15 mm/surface	3.6	2.3	30.3	
	[9]	Chemically etched to remove 0.125 mm/surface	4.0	1.6		
	[10]	Electropolished to remove 0.05 mm/surface	11.6	11.0		58.5

tested by Stack and Bobrowsky [6], is in all probability a nominal fracture stress also. The true fracture stress, that is the (fracture load/fracture cross sectional area) has also been calculated for all the samples investigated and these values are given in Table II.

4. Discussion

The scanning electron microtopographs shown in Fig. 2, reveal that the as-machined specimens of both materials contain surface cracks and/or fissures. These are removed by chemical etching or electropolishing post-machining treatments. However, chemical etching leaves what qualitatively appears to be a rougher surface finish than does electropolishing. In keeping with these observations, the mechanical test results for the as-machined samples are inferior to those given by either the chemically etched or electropolished test pieces. This is the case for both the bare and sheathed specimens. For the ingot material, the differences between the results for the chemically etched and electropolished specimens in either the bare or sheathed condition, are small and are probably not significant. However, the results for the hot-pressed powder material indicate that electropolishing enables significantly better tensile properties to be obtained than does chemical etching for specimens tested either bare or sheathed.

It may be concluded that for both grades of material investigated, sheathing gives a real enhancement of the mechanical properties as compared with those obtained by testing the specimens bare. This improvement is not so marked in the case of ingot material as with the powder beryllium, but even in the former case it appears to be significant. This is in agreement with the findings of Stack and Bobrowsky [6] but not with the work of Aladag *et al* [10].

Table III shows the mechanical test data obtained by other workers [6, 7, 9, 10] for bare specimens of extruded ingot and hot-pressed powder beryllium tested under a hydrostatic pressure of 78.7 kg mm^{-2}. These data are thus directly comparable with those obtained in the present investigation, as given in Table II. A comparison of the two tables shows that with the exception of the extruded ingot data of Aladag *et al* [10], all the other investigations have yielded mechanical properties lower than those achieved in the present work. However, in making such comparisons it must be borne in mind that there are many variables which may influence the results but which are difficult to quantify. These include grain size, purity, fabrication history and testing conditions. With regard to the ingot material, probably the most significant of these variables is the fabrication history. The production of beryllium bar by hot extrusion produces a marked texture in the material which increases the ductility of the material parallel to the extrusion axis. The increase in ductility generally increases with increasing extrusion ratio. Consequently, the disparity between the data of Aldag *et al* [10]

and that obtained in the present work, for the ingot material, is possibly attributable to differences in the degree of preferred orientation present in the beryllium samples used in the two investigations.

It is apparent from the above discussion, that in order to realize the best mechanical properties of beryllium in an hydrostatic environment, the specimens must be given an adequate post-machining treatment and their gauge sections must be sheathed. However, the effect of the sheath cannot be established from the present data. Stack and Bobrowsky [6] suggested that the effect of sheathing showed that beryllium exhibited a "Rebinder effect". The basis for this suggestion lies in the work of Likhtman *et al* [13] who demonstrated that many mechanical properties of both single crystal and polycrystalline metals are adversely affected by liquid environments containing organic polar molecules. In the present work, the methanol in the pressurizing fluid falls into this category of organic compound. Likhtman *et al* [13] did not demonstrate the effect of surface active media on tensile properties of the kind which have been measured in the present investigation. However, the present data may be interpreted on the basis that beryllium exhibits a Rebinder effect and that the sheathing acts as an impervious barrier which prevents the specimens being wetted by the surface active pressurizing fluid. The evidence is by no means conclusive and other explanations of the data are possible. For example, methanol slowly reacts with beryllium and the action of the sheath may simply be one of preventing such corrosion from taking place.

Acknowledgements

The authors are indebted to Mr J. H. Dingle for carrying out the optical metallography and to the Ministry of Defence for financial support for this work.

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Received 16 April and accepted 28 June 1973.