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SURFACE PREPARATION OF METALLOGRAPHIC SPECIMENS OF ZIRCONIUM-BASE ALLOYS CONTAINING COPPER

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OAK RIDGE NATIONAL LABORATORY
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J. E. Spruiell[1] and D. M. Hewette II

ABSTRACT

A combination of chemical and electrolytic polishing was successful in the metallographic preparation of Zr–1% Cu and Zr–1.6% Cu alloys. Chemical polishing produced flat surfaces that were suitable for examination with polarized light but were not suitable for examination with bright-field illumination. Electrolytic polishing applied to ground or mechanically polished specimens produced surfaces that were well etched but often were pitted heavily. A chemical polish followed by a short electrolytic polish produced microstructures suitable for examination with both bright-field and polarized light at high or low magnifications.

INTRODUCTION

An investigation was carried out to develop a reliable technique for preparing metallographic specimens of Zr–1% Cu and Zr–1.6% Cu alloys. This investigation was necessary, since the established polishing methods for the preparation of zirconium and zirconium alloys failed to produce the desired quality of microstructures in zirconium-copper alloys. This work was carried out as a part of a study of the kinetics of phase transformations of binary zirconium alloys.

APPLICATION OF ESTABLISHED TECHNIQUES

The conventional technique of mechanical polishing followed by etching was not successful in the preparation of metallographic specimens of zirconium-copper alloys. The cold-worked surface layer caused by the mechanical polishing

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[1]Consultant from the University of Tennessee.

led to nonreproducible appearances that were not characteristic of actual microstructural detail. This effect was evident even though mechanical polishing was done on a Syntron vibratory polisher.[2]

In the past, direct chemical polishing of as-ground surfaces has proved effective in the metallographic preparation of other zirconium alloys, but the polishing solutions that had been developed previously were not satisfactory for the preparation of zirconium-copper alloys. A chemical polishing solution was developed that was capable of yielding a flat but poorly etched surface suitable for polarized-light study but not for bright-field examination. The composition of this polishing solution is given below under the description of the adopted method. An example of the microstructure of a Zr–1.5% Cu sample obtained by using this chemical polishing solution is shown in Fig. 1. The martensitic-like phase was formed by quenching from a betatizing temperature of 1000°C. An unidentified transformation product formed at the grain boundaries and at the external surfaces of the sample on holding 60 min at 400°C. Note that very little detail is evident under bright-field illumination, and that it is difficult to differentiate between the two microconstituents.

Electrolytic polishing, utilizing perchloric acid–alcohol electrolytes, has proven to be applicable to metallographic preparation of zirconium and of zirconium alloys such as Zircaloy-2 (ref 3). However, this technique yielded pitted surfaces on zirconium-copper alloy specimens as shown in Fig. 2. The pitting occurred whether the specimen was as-ground or had been mechanically polished on the Syntron vibratory polisher.

APPLICATION OF COMBINED CHEMICAL AND ELECTROLYTIC POLISHING

From the results of the preceding experiments, it seemed logical to (1) apply chemical polishing in order to remove all distorted surface metal and produce a flat surface, and (2) follow with a short electropolish in order to obtain a well-etched surface.

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[2] R. J. Gray and E. L. Long, Jr., Preparation of Metallographic Specimens Through Vibratory Polishing, ORNL-2494 (Sept. 1958).

[3] D. M. Hewette II, Met. Div. Ann. Progr. Rept. July 1, 1960, ORNL-2988, p 388.

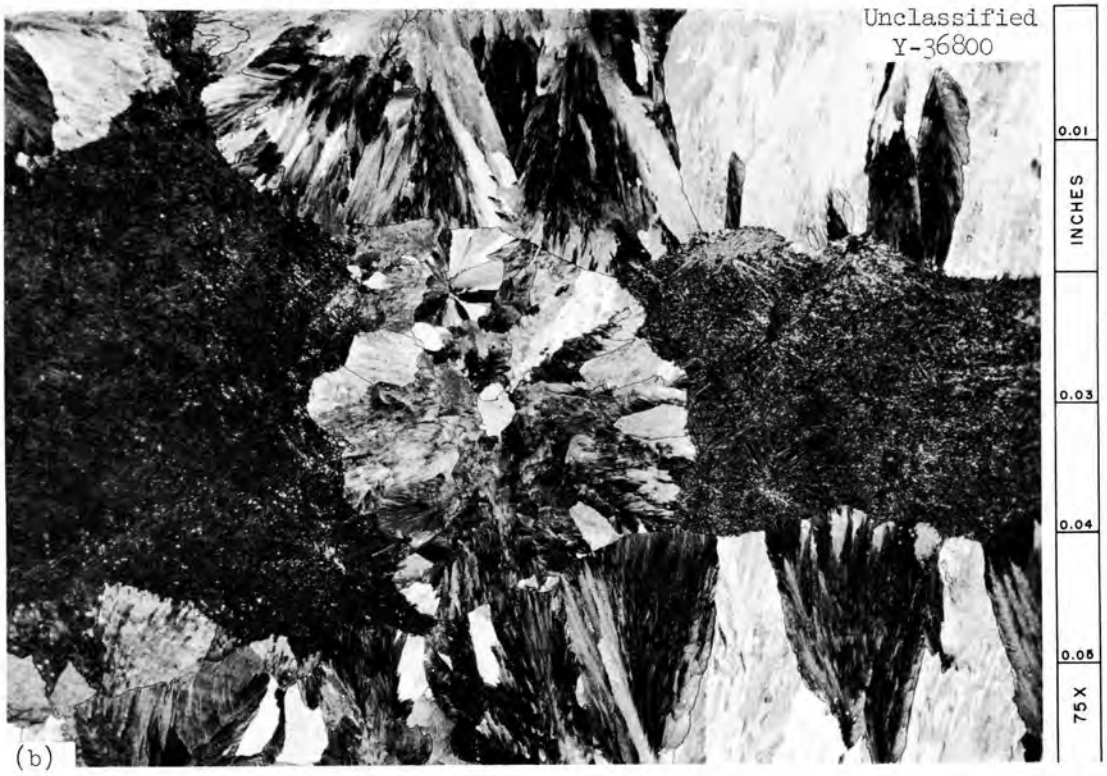

Fig. 1. Chemically Polished Zr-1.6% Cu. (a) Bright field, (b) polarized light. Little detail is evident under bright-field illumination. The microstructure is satisfactorily revealed under polarized light, although there is some "flowed metal" still evident. 75X.

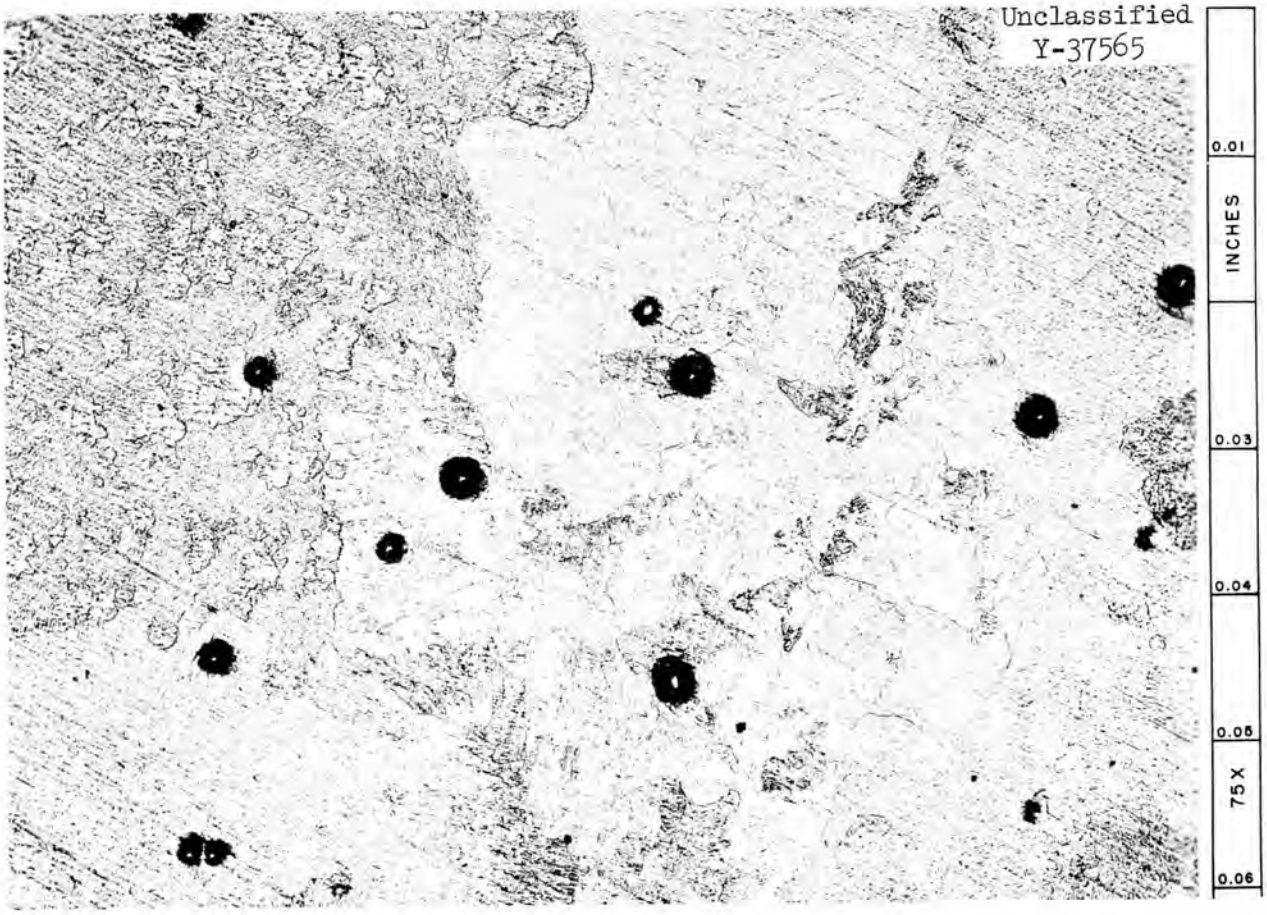

Fig. 2. Electrolytically Polished Zr-1.6% Cu. This sample was electropolished using a perchloric acid-alcohol electrolyte at 10 v for 40 sec. Pitting is evident, and yet not all the grinding scratches are removed. 75X.

The procedure began with pregrinding of the sample through 4/0 emery paper. After grinding, the sample was chemically polished with a solution of the following composition: 50 ml nitric acid, 50 ml lactic acid, 40 ml glycerin, 5–10 ml hydrofluoric acid, and 3–5 ml hydrochloric acid. The sample surface was swabbed vigorously with a cotton ball, which was well soaked with the polishing solution, until all distorted metal from grinding was removed, usually amounting to several thousandths of an inch. The sample was then rinsed in alcohol and, to avoid staining, immediately electropolished on a commercial electropolishing unit, the Disa-Electropol,[4] utilizing the following electrolyte: 200 ml perchloric acid, 100 ml butyl cellosolve, and 700 ml ethanol. The electropolishing was carried out for 8 sec at 8 v and a flow rate of 3 (corresponding to a pressure head of 9 cm).

Microstructures suitable for examination with either bright-field or polarized-light illumination and at low or high magnification have been obtained by using this technique. An example of a microstructure of a Zr–1.6% Cu alloy examined at a low magnification is shown in Fig. 3. This is the specimen shown in Fig. 1 and has the same products present. Notice that the details of the martensitic-like phase and the transformation product are revealed quite well under bright-field illumination and that there is also an excellent reaction of the specimen surface to polarized light. The advancing interface between transformation product and the martensitic-like microconstituent is shown at a high magnification in Fig. 4. Details of both microconstituents are resolvable by examination either with bright-field illumination or polarized light.

The quality of results obtained by using the chemical-electrolytic polishing technique was essentially the same for various heat treatments and for both Zr–1% Cu and Zr–1.6% Cu alloys.

CONCLUSIONS

A combination of chemical polishing and light electropolishing is suitable for the preparation of metallographic surfaces on Zr–1% Cu and Zr–1.6% Cu alloys. These alloys are not easily prepared for metallographic examination by standard methods.

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[4] Manufactured by H. Struer's Chemiske Laboratorium, Copenhagen, Denmark.

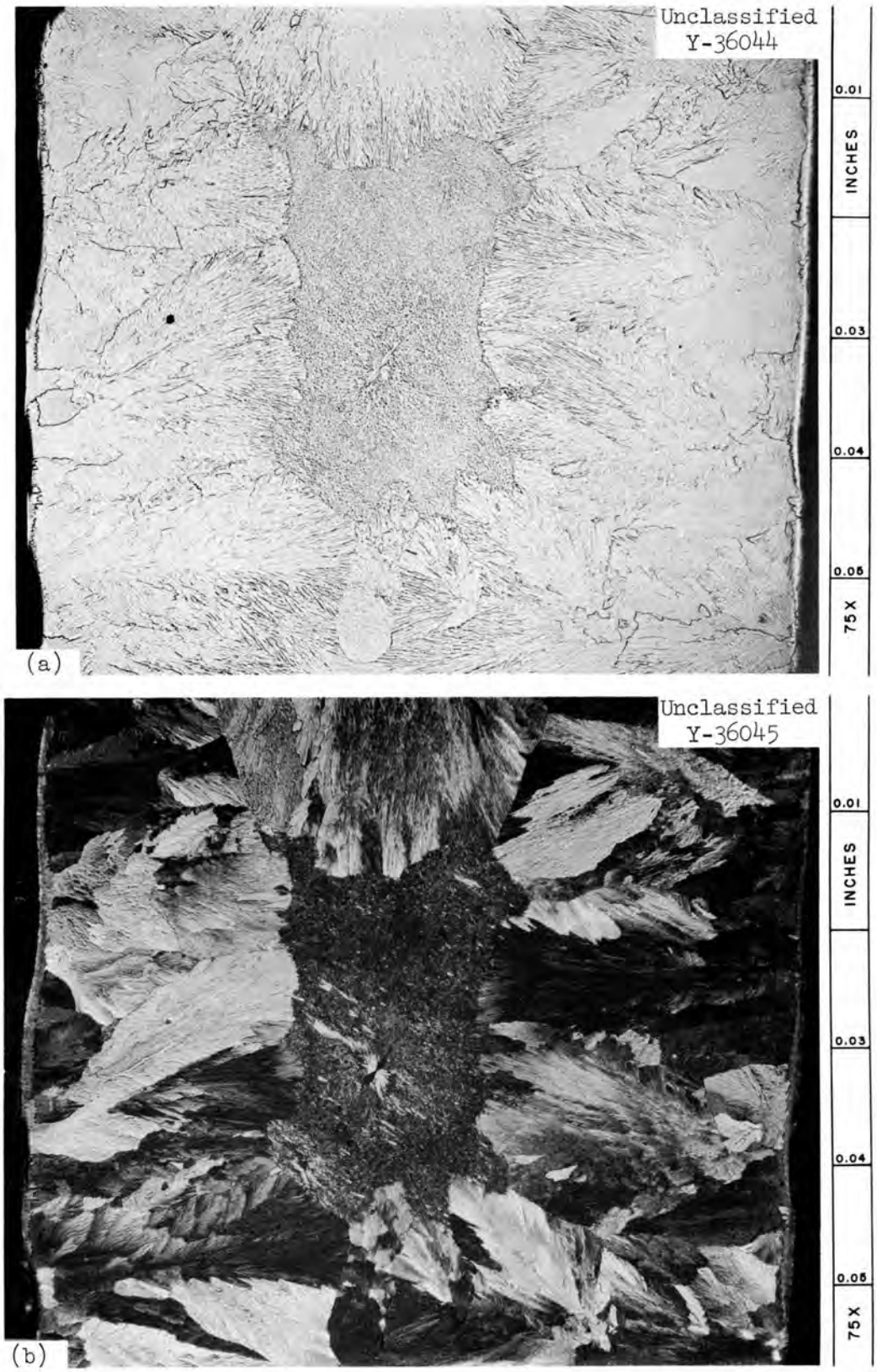

Fig. 3. Chemical-Electrolytically Polished Zr-1.6% Cu. (a) Bright field, (b) polarized light. 75X.

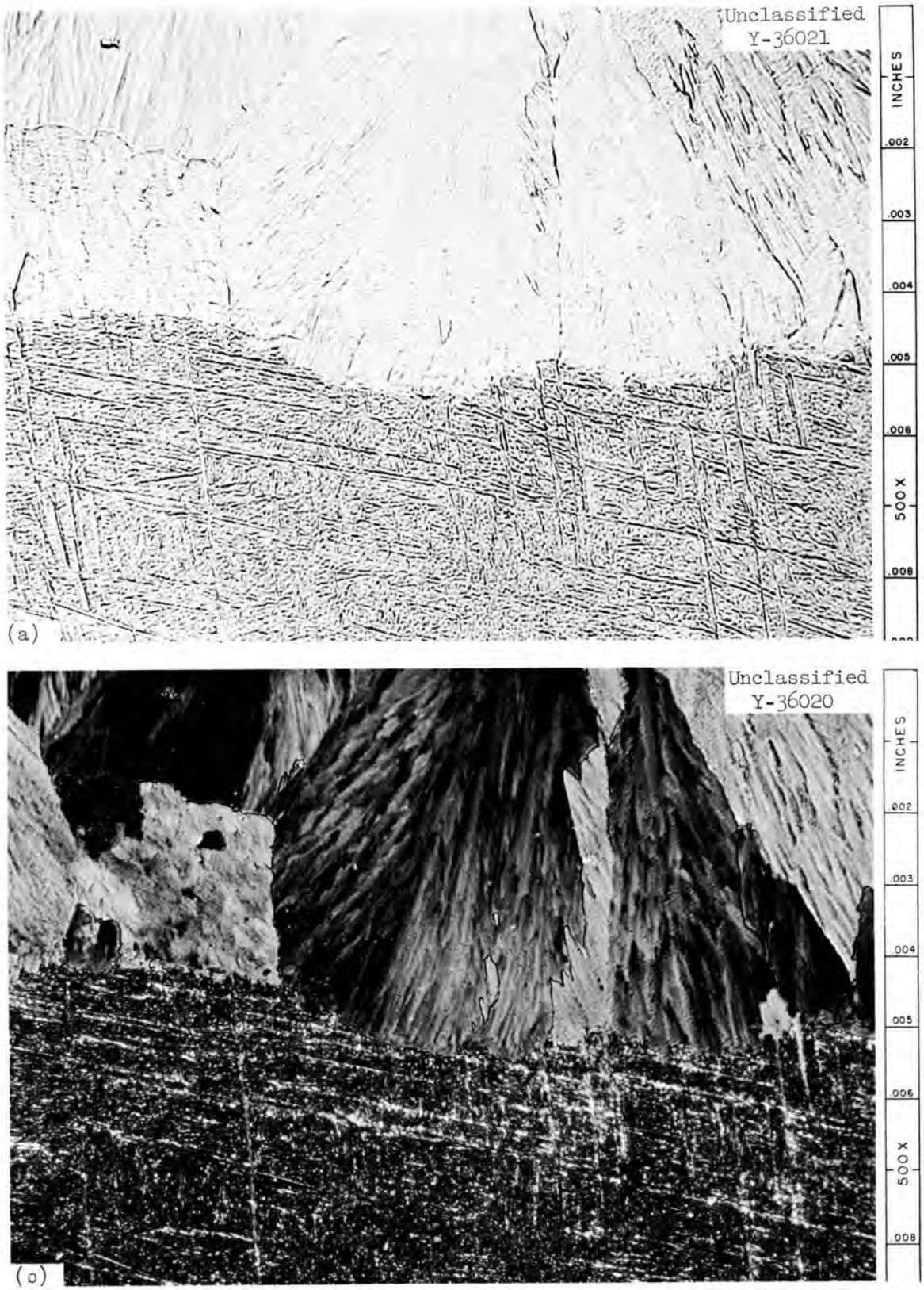

Fig. 4. Chemical-Electrolytically Polished Zr–1.6% Cu. (a) Bright field, (b) polarized light. 500X.

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