

cases, the cooperative reaction is quite possible outside the so-called coupled region and can very well occur there before the composition has moved into the coupled zone as a result of primary precipitation. The coupled zone should thus never be regarded as a region "only within which the alloy can solidify by a cooperative growth process".<sup>3</sup>

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## A Technique for Preparing Thin Foils of Ti and Ti Alloys for Transmission Electron Microscopy

R. A. SPURLING

Many techniques have been used in recent years for thin-foil preparation of Ti and Ti alloys. Both chemical<sup>1,2</sup> and electrolytic<sup>3-5</sup> thinning methods are currently in use, but the preference appears to be electro-thinning. The chemical thinning solutions generally are various concentrations of HNO<sub>3</sub>-HF while perchloric-acid containing solutions are used for electro-thinning. Cooling the solutions (chemical or electrolytic) is the key to successful foil making of Ti and Ti alloys because of a better controlled polishing rate and a minimization of H<sub>2</sub> pick-up.<sup>3,6</sup> Consistency in thinning single phase ( $\alpha$  or  $\beta$ ) alloys using the existing thinning solutions can be maintained but when thinning two phase alloys one of the phases generally is preferentially attacked. The technique described below was developed for thinning two phase ( $\alpha + \beta$ ) structures and is equally efficient for either  $\alpha$  or  $\beta$ -phase samples. The electrolyte has the advantage over other solutions in that it polishes more rapidly and is less hazardous than the perchloric acid solution.

Specimens to be thinned are secured to a flat block by an alcohol-soluble wax, then mechanically ground through 600 grit SiC abrasive to a thickness of 0.0025 cm. Care must be taken in handling specimens in this step so as not to introduce deformation. Thin foils

are then made using the window method with an acetone-soluble lacquer to mask edges of the specimen. An electrolyte consisting of 5 pct H<sub>2</sub>SO<sub>4</sub> by volume in methanol is the final thinning solution. Conditions for preparing thin foils are listed in Table I. The electrolyte is stirred continually during the thinning process, while the specimen (anode) remains 2 to 3 cm away from the platinum cathode.

After thinning, the specimen is washed in methanol. Because of a residue that forms on some of the foils, the following cleaning procedure is needed. From the methanol the foil is washed in H<sub>2</sub>O a few seconds, then given a 30 sec wash in concentrated HNO<sub>3</sub> acid. Upon removal from the acid the foil is washed in H<sub>2</sub>O, then methanol and finally immersed in ethyl alcohol during cutting to E.M. size specimens.

Figs. 1, 2, and 3 are typical micrographs of foils made using the above technique. Compositions of the alloys are expressed in wt pct. Fig. 1 is a micrograph

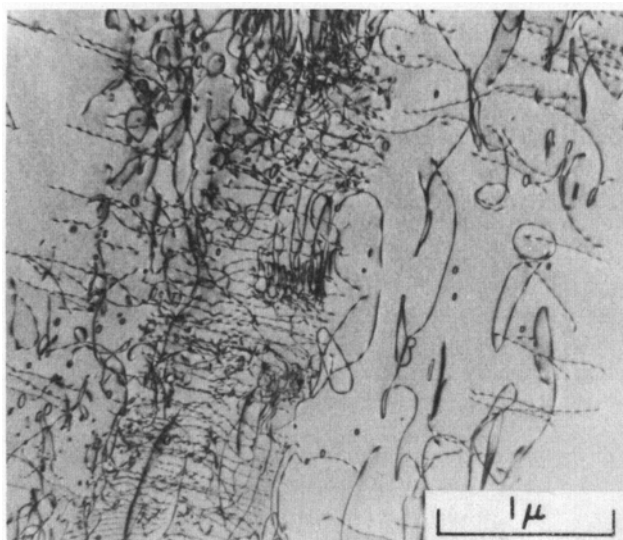


Fig. 1—Transmission electron micrograph of Ti-5Al single crystal annealed at 1125 K and strained 2 pct at 600 K.

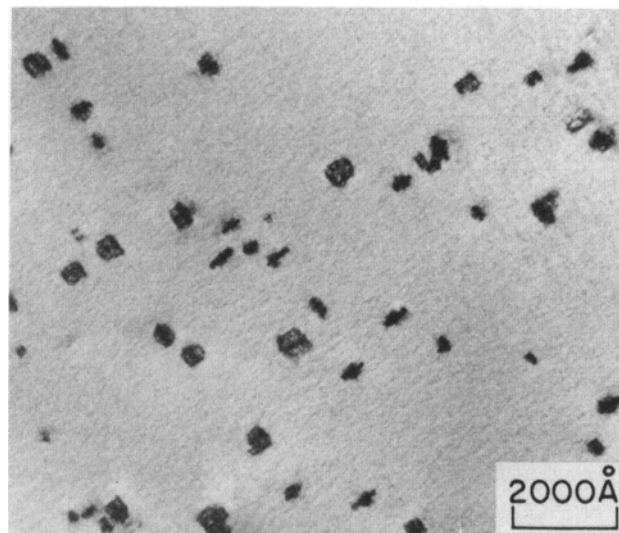


Fig. 2—Transmission electron micrograph of Ti-40V + 250 ppm O<sub>2</sub> solution treated and aged 675 K/1 week.

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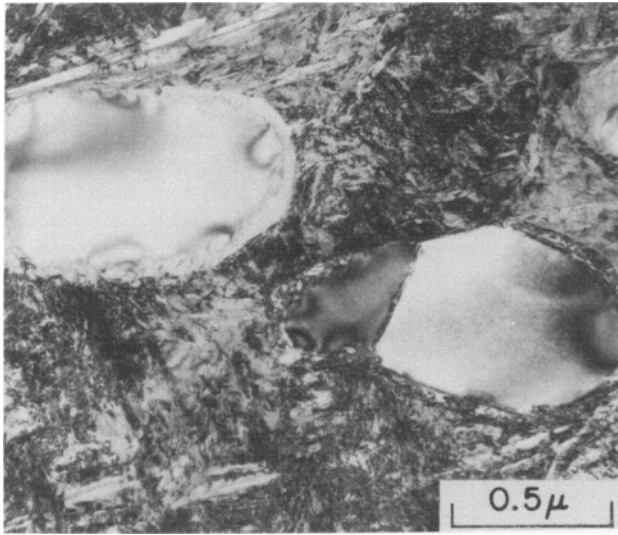


Fig. 3—Transmission electron micrograph of Ti-6 Mo-3 V-4 Zr-2.5 Al solution treated and aged 785 K/8 h.

Table I. Electro-Polishing Conditions for Preparation of Thin Foils

Alloy Type	Open Circuit Voltage	Current Density* mA/cm <sup>2</sup>	Temperature of Electrolyte (K)
$\beta$	8	110	223
$\alpha$	12	170	223
$\alpha + \beta$	12	150	238

\*Typical specimen size is 0.5 cm<sup>2</sup>.

of the  $\alpha$  phase alloy Ti-5 Al single crystal showing a  $\{10\bar{1}0\}$  slip band containing  $\bar{a}$  type or  $1/3 \langle 11\bar{2}0 \rangle$  dislocations. Fig. 2 is a micrograph of a Ti-40 V containing 250 ppm O<sub>2</sub> showing  $\beta'$  precipitation in a  $\beta$  matrix. Here the  $\beta$  phase has not undergone spontaneous relaxation,<sup>3</sup> a deleterious thin-foil effect which frequently occurs during thinning. In those alloys where the spontaneous relaxation distorts the microstructure, it can be avoided by ion thinning.<sup>7</sup> Fig. 3 is a micrograph of Ti-6 Mo-3 V-4 Zr-2.5 Al alloy showing primary  $\alpha$  particles in a transformed  $\beta$  matrix. There is no preferential polishing in the two phase structure. This micrograph best illustrates the quality of the foils that can be prepared using this technique.

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## Discussion of "Thermodynamics of $\beta_1$ -NiZn and $\alpha$ -NiZn Phases"

R. P. ANANTATMULA

In a recent communication to this journal, Kou and Chang<sup>1</sup> compared the activity data of zinc in  $\beta_1$ -NiZn of Anantatmula and Masson<sup>2</sup> with the theoretical equations derived by Gyuk, Liang and Chang<sup>3</sup> for face-centered tetragonal (fct) lattices, and obtained excellent agreement for a value of  $\alpha = 0.0004$ . Although the lattice of  $\beta_1$ -NiZn is fct of the AuCu type, Anantatmula and Masson used the equations for CsCl-type alloys derived by Chang *et al.*<sup>4,5</sup> for a comparison of the theory with experimental activity data. The agreement was very good for an  $\alpha$  value of 0.0003. As pointed out by Kou and Chang, in general, one cannot use the equations of CsCl-type alloys in the case of AuCu-type alloys. This is because the nearest neighbor arrangement is different in the two types of lattices, which leads to differences in bonding energy and consequently the enthalpy terms will be quite different. However, the use of the CsCl-type equations by Anantatmula and Masson in comparing the theory with experiment is justified in the case of  $\beta_1$ -NiZn due to the following.

In Fig. 1 is reproduced a schematic of two fct unit cells of the  $\beta_1$ -NiZn phase. The atoms represented by the hollow circles are nickel atoms and solid circles represent zinc atoms. This structure may also be reproduced by a repetition of body-centered tetragonal (bct) unit cells. The unit cell represented by the dark lines is the bct cell with an axial ratio different from that of the fct unit cell, since the 'a' parameter is different. Let us now examine the nearest-neighbor configuration of this structure. We have two different tetragonal sublattices represented by nickel and zinc atoms, respectively. The nearest-neighbor arrangement can be deduced by considering the zinc atom P in the center of the nickel sublattice.

We have

$$AG^2 = AC^2 + CG^2 = 2a^2 + c^2 \quad [1]$$

Therefore,

$$AP = \frac{1}{2}AG = \frac{1}{2}\sqrt{2a^2 + c^2} = \frac{a}{2}\sqrt{2 + \frac{c^2}{a^2}} \quad [2]$$

This means that all the atoms on the nickel sublattice are at a distance  $\frac{a}{2}\sqrt{2 + \frac{c^2}{a^2}}$  from the zinc atom P.

But the atoms that are closest to the zinc atom P on the zinc sublattice are at a distance 'a' from it. Therefore, in the ideal case of twelve nearest neighbors, we should have

$$\frac{a}{2}\sqrt{2 + \frac{c^2}{a^2}} = a \quad [3]$$

or

$$\frac{c}{a} = 1.414 \quad [4]$$

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