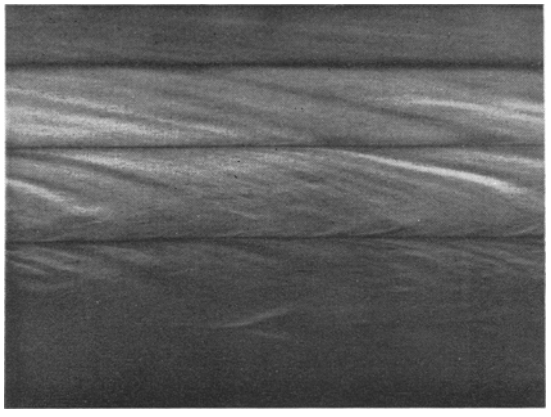

Figure 2 "Chevron" type markings in deformation bands.

some anomalous effect of the machining. In addition, no feature of the experimental apparatus was found that could cause 20° pinning or any multiple of 20° pinning. It became clear that the band formation was a feature of the material itself. The cause of the regular band formation was eventually revealed by a technique which might be called "metallographic".

Samples of the raw unmachined extruded rod were machine faced, polished, and finally etched in acetone. After etching, a regular hexagonal honeycomb structure was observed on the cross-section of the extruded rod (Fig. 3). Subsequently, similar features were revealed by acetone-etching on undeformed tube specimens cut from the rod (Fig. 1b). Further enquiries revealed that this regular structure was produced during the initial extrusion process by extruding the rod through a multi-holed breaker plate.

The macroscopic structure generated in the raw material during production, as revealed by the polishing and etching techniques, obviously

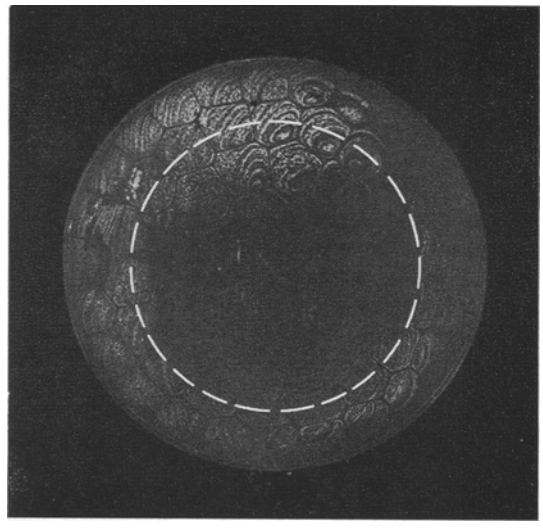

Figure 3 Etched surface of raw PVC rod; dotted line indicating position from which specimens are machined.

led to the deformation mode observed during testing. This observation highlights the necessity to characterize the test specimens not only for molecular-structural properties (for example, molecular weight distribution) but also macroscopic structures. The polishing and etching technique, which has proved of immense value in metallurgical studies, may also be of use in rheological investigations of plastics.

Received 30 August
and accepted 20 November 1972

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Reaction zones in Ti/W composites

This letter describes microscopic, kinetic, and microhardness observations of solid-state interfacial reactions between tungsten filament and titanium, made as part of an experimental study on metal-matrix composites. The solid-state region of the titanium/tungsten system is characterized by a β eutectoid decomposition at 715°C that forms two phases of tungsten and α titanium solid solutions [1].

Composite specimens were prepared using 0.2 mm commercial lamp-grade W filament and 0.41 mm thick sheets of commercially pure Ti75A. These components were diffusion bonded at 1000 psi (6.9 MNm^{-2}) for 1 h at 875°C under 10^{-4} mm Hg vacuum. Reaction-zone studies were carried out by vacuum annealing in the temperature range 760 to 930°C and by furnace cooling to produce measurable reaction zones.

On slow cooling from the diffusion annealing temperature, the Ti/W interfacial region forms

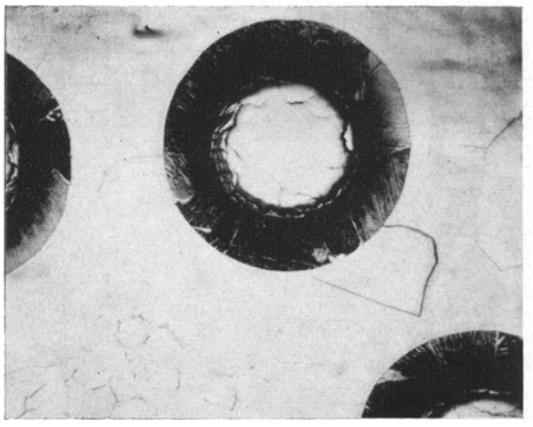

Figure 1 Tungsten filaments in titanium matrix after 870°C/100 h diffusion anneal and furnace cooling; specimen surface polished plus light Kroll's etch (× 60).

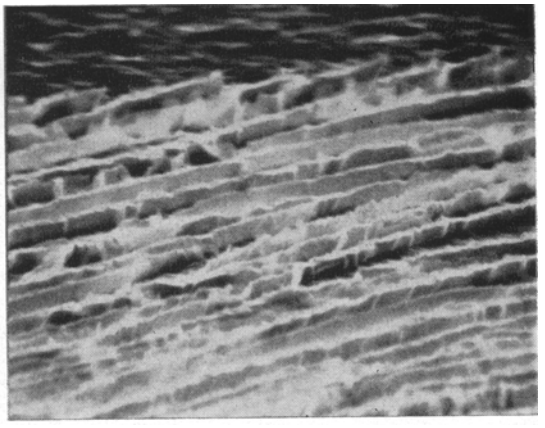

Figure 3 Platelets of lamellar region showing W solid solution constituent standing in relief after light etch (× 8000).

a lamellar eutectoid reaction product of α-Ti and W solid solutions that encases the W filaments, as shown in Fig. 1. The dark region adjacent to the filaments consists of a fine-plate structure that merges into a finer acicular inner zone as shown in Fig. 2. The phase within the eutectoid platelet structure is seen to be continuous with the outer surrounding α titanium matrix. The chiseled parallelepiped morphology of the W plates standing in relief after etching is shown in Fig. 3. The platelet spacing decreases with increasing W concentration as the filament peripheral region is approached, where the structure probably consists of a combination of blocky and Widmanstätten proeutectoid W in transformed β titanium. It has been noted that at very high W concentrations, such as the condition of the filament edge, the $\beta \rightarrow \alpha$ transformation is very sluggish and may not readily occur [2]. In this work, X-ray diffraction showed patterns only for α titanium and tungsten solid-solution. An electron microprobe scan for W showed a steep drop in W concentration at the edge of the eutectoid structure and indicated a low-concentration W diffusion zone in the surrounding α titanium matrix; corresponding W concentrations from the Ti-W phase diagram are ~ 28 and ~ 0.8 wt %, respectively.

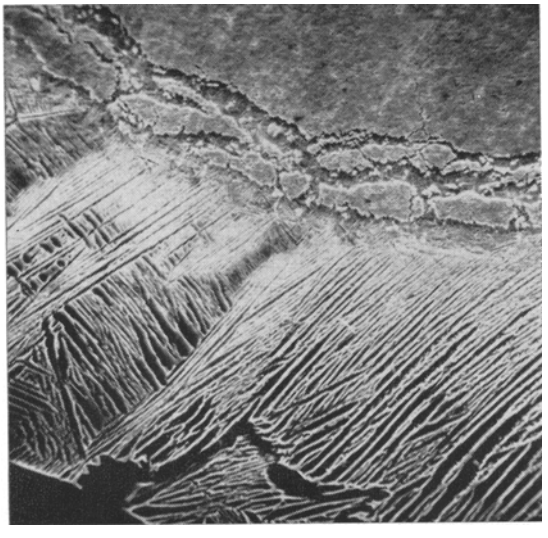

Figure 2 Ti/W interfacial region (870°C/100 h) showing transition from widely-spaced lamellar structure at eutectoid outer edge to fine acicular constituent at filament periphery. Polished plus light Kroll's etch (× 800).

The growth of the eutectoid reaction zone as a function of time is shown in Fig. 4. Logarithmic plots of these slopes versus inverse temperature indicate the applicability of an Arrhenius relationship, as seen in Fig. 5. A linear temperature-dependence of the reaction is noted along with a break in the curve at ~ 870°C, which is lower than the expected value of ~ 950°C for the α/β transus of this Ti alloy. It is likely that the break reflects the lowering of the β transus as a result of W diffusion. The measured activation energy above the break (obtained by multiplying the slope by two, converting K to $cm^2\ sec^{-1}$) was ~ 50 kcal mol^{-1}, which agrees with a reported value of ~ 49 kcal mol^{-1} for W diffusion in β Ti [3].

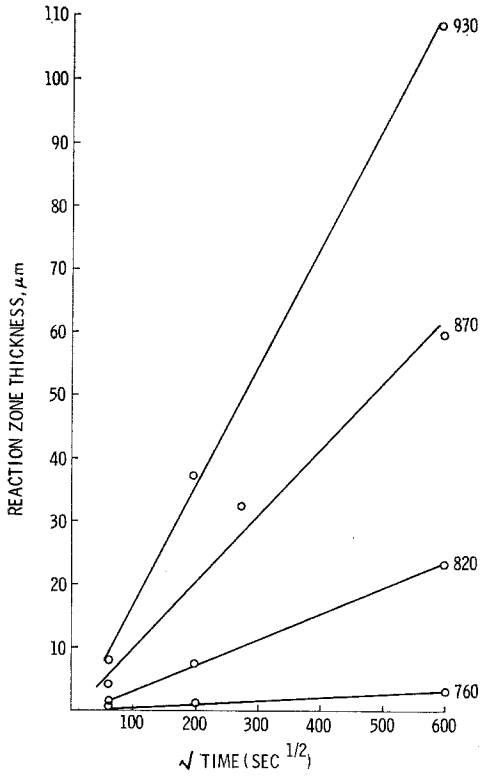

Figure 4 Growth of Ti/W reaction zone as a function of time at temperature.

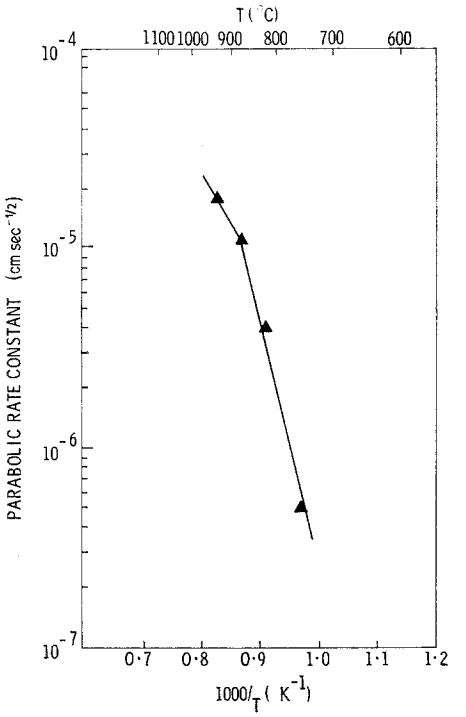

Figure 5 Temperature dependence of rate constant for eutectoid growth at Ti/W interface.

A measured activation energy of ~ 117 kcal mol^{-1} below the break closely agrees with ~ 126 kcal mol^{-1} for W self-diffusion [3].

Vickers microhardness tests were made on all reaction specimens that had measurable hardness zones. Interestingly, it was found that the eutectoid reaction zone was softer than the adjacent diffusion zone and matrix material, suggesting that a relatively tough transition material exists between filament and matrix. This effect is shown schematically in Fig. 6 and represents the typical trend of average hardness values. In subsequent tensile tests on Ti/W composites with eutectoid reaction zones up to ~ 65 μm wide it was found that no significant changes in strength could be attributed to zone thickness. Examination of the tensile fracture region showed a high degree of interfacial bonding and intact reaction zones between the Ti and W with no observable debonding, indicating that the Ti/W solid solutions may offer enhanced shear resistance in such composites.

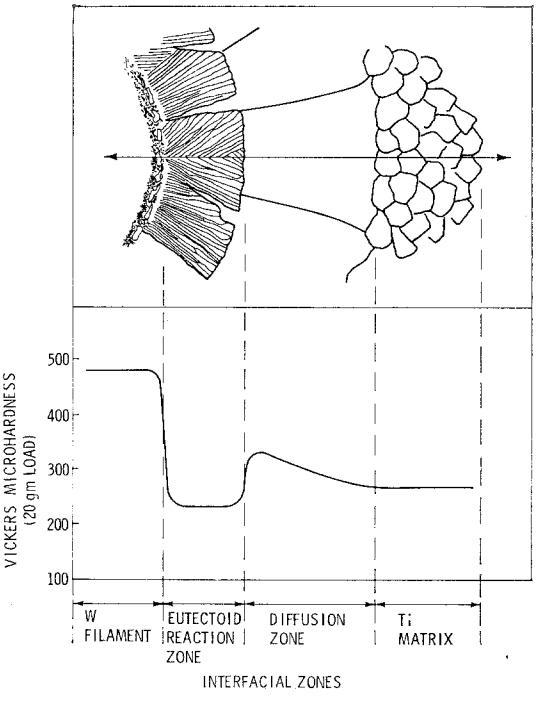

Figure 6 Schematic of typical microhardness across Ti/W interface. Average hardness of various conditions is represented.

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Received 9 October
and accepted 16 October 1972

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Improved visibility in crystal pulling

In the field of Czochralski growth of crystals [1], one of the prerequisites for controlled crystal growth is clear visibility of the solid-liquid interface. The technique of liquid encapsulation [2, 3], though primarily designed to prevent loss due to volatilization, has gone some way towards alleviating this problem. However, in the growth of some crystals, for example PbTe and GaAs, some material is still lost during the growth run. This material comes from the seed or from those parts of the crystal which are not covered with encapsulant, from the melt by a path between the encapsulant and the crucible or crystal caused by inadequate wetting by the encapsulant,or by the transport of vapour from the melt in bubbles. This leads to a certain amount of vapour deposition on the cooler parts of the surrounding chamber and window, leading in time to reduced visibility, and even, in the case of some very prolonged runs, a complete loss of visibility.

The problem can be tackled in one of two basic ways; by allowing the volatile material to deposit on the window and then removing it, or by preventing the material from depositing on the window in the first place. Our first approach was to use the former method, and to this end a window-wiping arrangement was devised which would clear the window of deposited material. This is shown in Fig. 1. The device comprises steel wool wrapped around a spring bronze arm, which is clamped to a metal shaft by a metal boss and two holding bolts. The shaft passes through a hole, drilled with a diamond drill, at the centre of a normal $2\frac{1}{2}$ in. silica window, as fitted to the standard RRE puller. It has an O-ring seal fitted on either side of the window, so that the growth chamber can be operated with either a positive or a negative pressure, with respect to the laboratory. A knurled knob is fitted to the outer end of the shaft to facilitate rotation of the wiper. Whenever viewing of the crystal becomes difficult the wiper is rotated, and the window brushed clean by the steel wool. A single rotation is usually sufficient.

The second system was developed to solve the problem of deposited material which is more tenacious, and hence cannot be easily wiped off the window, and also the problem of corrosive vapours, such as the As vapour above GaAs, which might attack the components of the window wiper. This assembly is an adaption of the window arrangements used in the RRE High Pressure Puller Chamber [4]. The window assembly is shown in Fig. 2, and con-

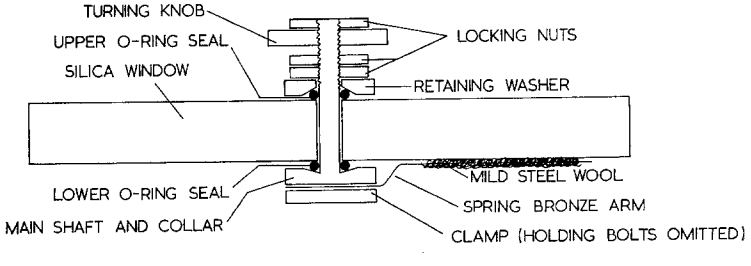

Figure 1 Window wiper assembly.