

## EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE OF V-4Cr-4Ti MATERIALS

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### OBJECTIVE

The objective of this task is to determine whether the banded structure observed in an extruded V-4Cr-4Ti bar from ANL's 832665 heat and in a swaged rod from the General Atomic (GA)'s 832864 heat can be effectively removed by dissolution at elevated temperature.

### SUMMARY

A banded structure containing Ti-rich particles has been observed in an extruded V-4Cr-4Ti bar from the ANL's 832665 heat and in a swaged rod from the GA's 832864 heat [1]. Because these two materials may be used as feedstocks for the upcoming creep tubing fabrication, this task was undertaken to determine whether this secondary-phase structure could be removed by dissolution at elevated temperature. Small pieces of the two materials were heat treated at 1150°C for 3 h and then characterized. The preliminary results show that this heat treatment did not redissolve the banded structure in either the extruded 832665 bar or the swaged 832864 rod.

### EXPERIMENTAL PROCEDURE

A rectangular bar of 2.5 x 1.2 x 1.0 cm from ANL's 832665 heat and a rod section of 1.0 cm diameter x 1.2 cm long from GA's 832864 heat were used in the heat treatment. Before the annealing, the specimen surfaces were carefully prepared to remove impurities introduced in specimen cutting. The preparation process included grinding with 600-grit silicon carbide paper, polishing with 6- $\mu\text{m}$   $\text{Al}_2\text{O}_3$  or diamond powder, and then cleaning with acetone and methanol. The specimens were then wrapped in a Ti getter foil and annealed in a high vacuum ( $>3 \times 10^{-7}$  torr) at 1150°C for 3 h, as suggested by Rowcliffe and Hoelzer [2].

Following the heat treatment, metallographic specimens were cut perpendicular to the extrusion direction of the bars to elucidate the microstructure, particularly the distribution of the banded structure. After mechanical polishing and electropolishing (see Fig. 1), optical microscopy was used to delineate grain morphology and microstructural inhomogeneity.

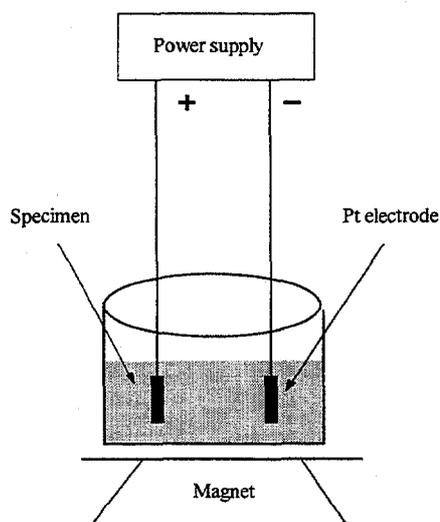


Fig. 1. Schematic diagram of electropolishing equipment

## RESULTS AND DISCUSSION

As reported previously [1], there were significant microstructural differences, including grain sizes, between the extruded 832665 and the swaged 832864 materials. The microstructure of the extruded 832665 material consists of an inhomogeneous mixture of small (5-20  $\mu\text{m}$ ) and coarse grains (25-50  $\mu\text{m}$ ). The grains are mostly equiaxed. In the swaged 832864 material, the grains are elongated in the longitudinal direction and the average grain size is larger than that of the 832665 material. In both materials, banded structure parallel to the work direction was observed before the heat treatment, as shown in Figs. 2a and 3a [1].

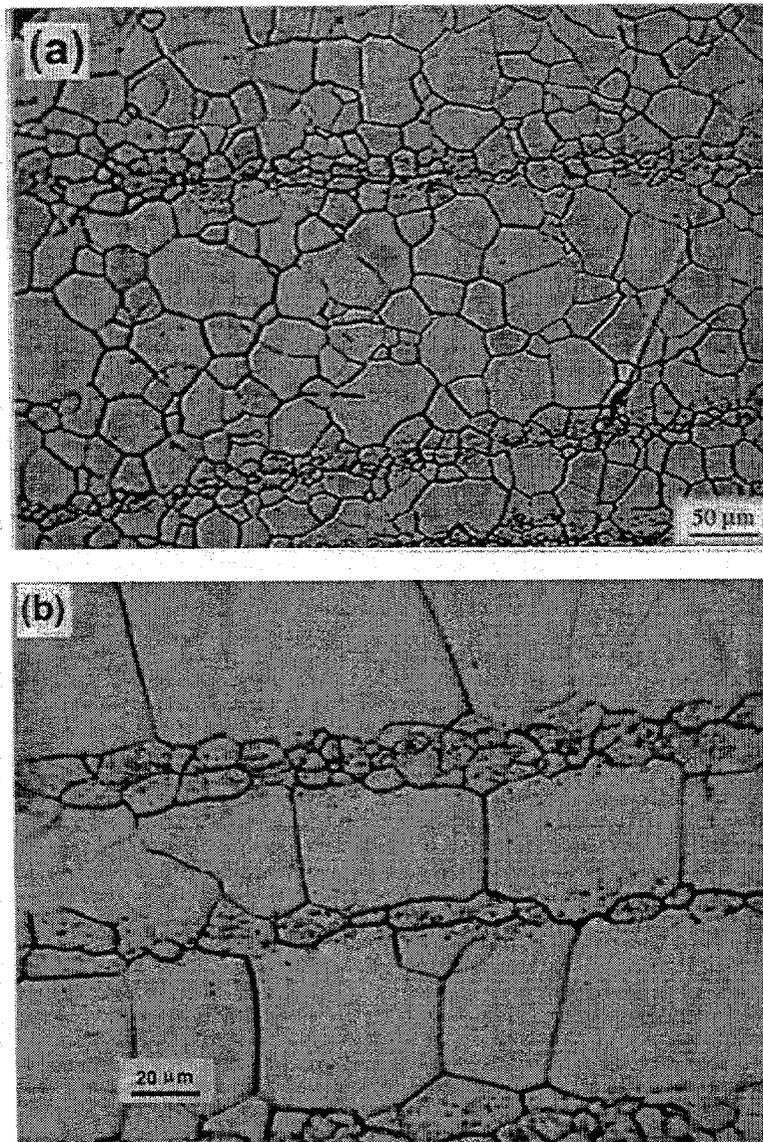


Fig. 2. Micrographs of local area with banded structure in extruded 832665 material (a) before and (b) after the heat treatment.

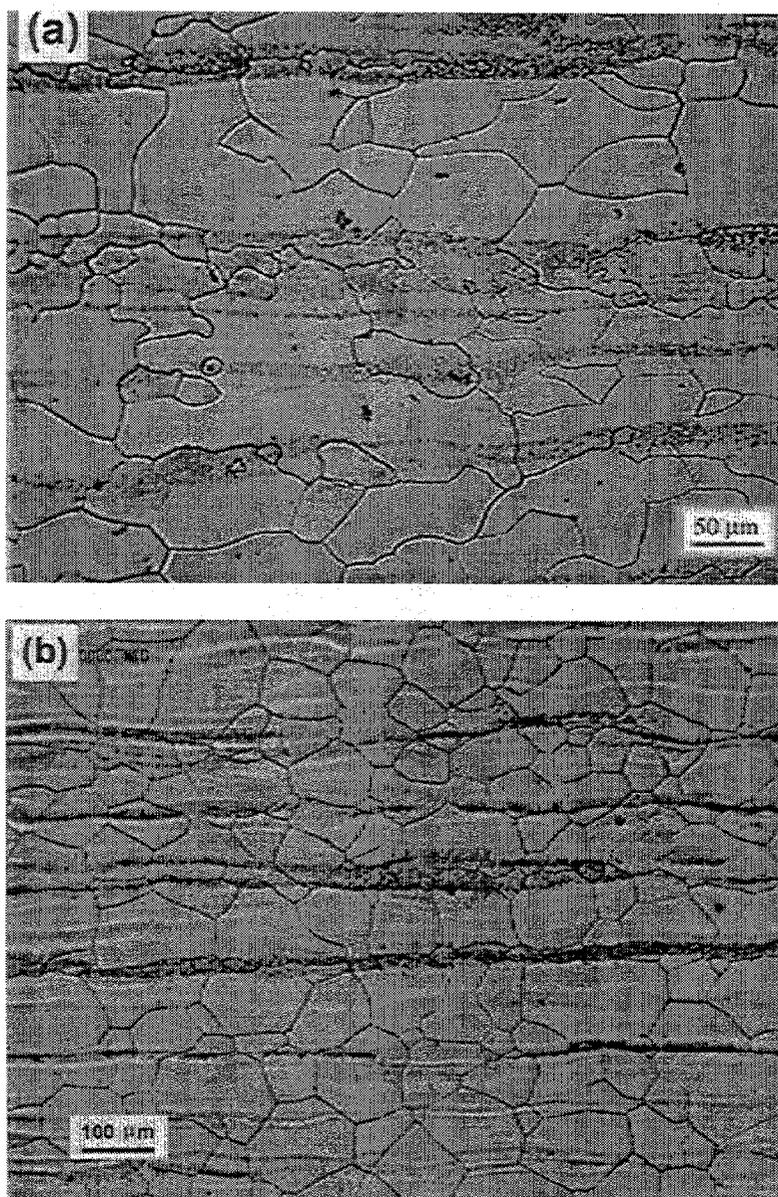


Fig. 3. Low magnification micrograph of banded structure in heat-treated 832864 materials (a) before and after (b) heat treatment.

Metallography after the heat treatment showed that the 1150°C annealing did not redissolve the banded structure in either the extruded 832665 bar or the swaged 832864 rod. Figure 2(b) shows a local area with the banded structure in the extruded 832665 material after heat treatment. Similar to the as-received specimens, Fig. 2(a), the average grain size in the banded region is considerably smaller than that in the unbanded region. Detailed analyses indicate that the width of the bands and the spacing between them vary from region to region. The observed width of the bands in the post-heat-treatment specimens ranges from 25 to 75 μm (pre-heat-treatment specimen: 25 to 100 μm), and the distances between the bands range from 100 up to a few hundred μm (pre-heat-treatment specimen: ≈ 50-150 μm) in the regions examined.

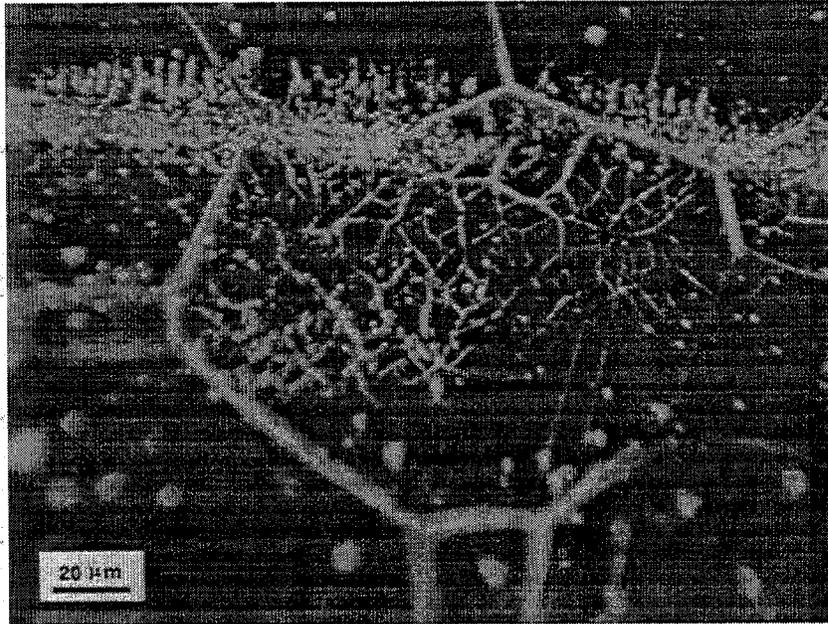


Fig. 4a. "Branched" structure associated with Ti-rich secondary particles within band structure in heat-treated 832864 materials after heat treatment.

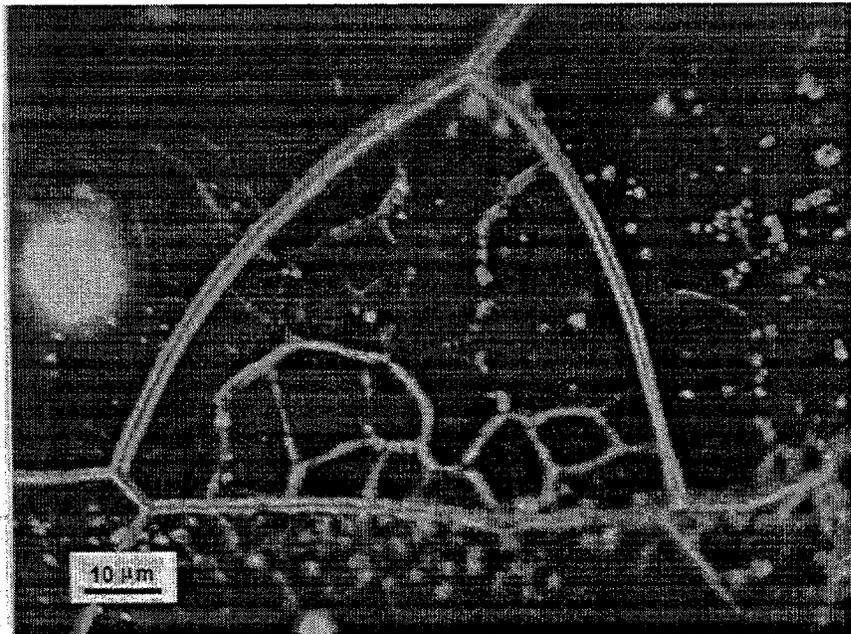


Fig. 4b. Subgrains in original grain of heat-treated 832864 materials.

Fig 3(b) is a lower-magnification micrograph of the banded structure in the heat-treated 832864 material. For comparison, a micrograph of the as-received specimens is shown in Fig. 3(a). It appears that the heat treatment did not dissolve the banded structure although it did cause the previously elongated grains to become more equiaxed. As in the 832665 material, Fig.3(b) shows that the grain size in the banded region is smaller than in the unbanded region, and the width of

the bands and the spacing between them are also varied from region to region. A new characteristic microstructure was observed in higher-magnification micrographs of swaged 832864 material after the heat treatment. Fig. 4(a) shows a 'branched' structure associated with the Ti-rich secondary particles in the banded structure. The branches are apparently formed by joining associated with secondary particles. Directions of the branches are mostly parallel to the faceted interfaces of the grain, which is usually a low-index plan of the material. In addition, subgrains are found within grains of the heat-treated 832864 materials, as shown in Fig. 4(b). Subgrain size ranges from a few  $\mu\text{m}$  to 15  $\mu\text{m}$  and there is a strong tendency for the subgrain boundary interfaces to be faceted similarly to those of the original grain surfaces. The branched structure appears to be related to dissolution and diffusion of secondary particles. More detailed study is required to completely understand the mechanism of such structure.

#### **FUTURE ACTIVITIES**

In addition to the extruded bar and swaged rod, a piece of rolled 3.8-mm-thick plate from the 832665 heat has been heat treated under similar conditions. The microstructure of this plate material will be characterized and compared with that of the untreated material. Result will be presented in a future report.

#### **REFERENCES**

1. Y. Yan, H. Tsai, and D.L. Smith, Fusion Materials Semiannual Progress Report DOE/ER-0313/27, Dec. 1999.
2. F. Rowcliffe and D. T. Hoelzer, Fusion Materials Semiannual Progress Report, DOE/ER0313/25, Dec. 1998, pp. 42-58.