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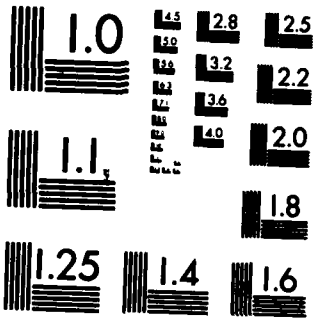

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TECHNICAL REPORT

OFFICE OF NAVAL RESEARCH

Contract USN 00014-75-C-1012

X-Ray Topography of Hydride Domains

by

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April 1983

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ABSTRACT

X-ray topographic techniques were applied to the study of twin related domains in niobium hydride and deuteride cyrstals. It was shown that contrast between the domains results from the small differences in orientation. The procedure has the advantages of not requiring as careful surface preparation as required by optical methods and of being a relatively rapid technique. The method can be applied to any of the single crystal hydrides.

1. INTRODUCTION

The attention focused on metal-hydrogen systems in recent years has resulted in the development of a number of techniques for examining the structure and morphology of the hydride phases (Schober and Linke 1976a). The orthorhombic β-NbH phase has been shown to consist of six twin-related orientations of the hydride which derive from the ordering of the hydrogen interstitials in the original b.c.c. structure. Schober and Linke (1976b) and Birnbaum et al (1976) have used optical microscopy with polarized light to reveal the inter-relationship between differently oriented domains of the hydride phase and the domain structure has also been examined with TEM (Schober 1975). Based on these observations, it appears that arrays of narrow twin-related domains are energetically more favorable than large blocks of a single orientation. While both the optical metallography and TEM have proven to be useful techniques to elucidate the hydride structures, they require extensive specimen preparation prior to viewing the domains.

2. EXPERIMENTAL

The present work describes the use of x-ray diffraction topography to image domains in β-NbH and β-NbD crystals. Relatively little surface preparation was required, aside from an ordinary metallographic polish. The

time required to produce a topograph on dental film with a double crystal camera and with radiation from a conventional sealed tube source was less than one hour.

Niobium crystals grown by a strain-anneal technique were spark machined to produce (110) surfaces which were then mechanically polished. Hydride and deuteride crystals having the compositions $NbH_{0.78}$ and $NbD_{0.75}$ were grown by slow cooling of the niobium crystal from 700°C under controlled pressures of hydrogen and deuterium gases, respectively. X-ray topographs were taken of the niobium-hydride (deuteride) crystals using asymmetric crystal topography (ACT), a variant of double crystal topography (Boettinger et al 1976), in the non-dispersive Bragg configuration. Sealed tube x-ray sources having spot focuses were used to provide the characteristic Mo K_α or Cu K_α radiation. The $\underline{g}$ = [220] diffraction vector was used for the reflection topographs which were recorded on Ilford L4, 50 μm thick nuclear emulsions, or on Kodak double sided DF-57 dental film. The former provides much better resolution whereas the latter requires considerably less exposure for adequate contrast (exposures of about 45 minutes were required using Mo K_α radiation produced at 45 kV with a tube current of 20 mA).

3. RESULTS AND DISCUSSION

Back reflection Laue photographs of the $NbH_{0.78}$ and $NbD_{0.75}$ crystals showed splitting of the diffraction spots which was not present before the introduction of hydrogen or deuterium and which indicates the formation of a domain structure within the crystals. The spatial arrangement of domains in a deuteride crystal is seen in the optical micrograph of the $NbD_{0.75}$ crystal (Fig. 1a) and in the corresponding reflection topographs (Figs. 1b and c). As shown by the optical micrograph (Fig. 1a), which was taken with oblique illumination, the structure of the hydride crystal consists of sets of domains

arranged in a plate morphology. Each region, such as those marked A - F, contains at least two sets of domains. This structure has been shown (Schober 1975, Schober and Linke 1976a and b, Schober and Wenzl 1976, Birnbaum et al 1976), to consist of sets of parallel, twin related domains or regions where H ordering on subsets of interstitial sites has produced elastic distortions of the cubic structure such that the resulting orthorhombic structures are twin related. In the optical micrographs the contrast is due to the surface tilts associated with the twinning. (Similar contrast may be obtained using polarized light and results from the optical anisotropy of the orthorhombic structure).

Domain structures, identical to those seen in the optical micrographs, were obtained using reflection topography as shown in Figs. 1b and 1c. In this case the contrast is due to the variation of the diffraction conditions between the different domains. A rotation of the specimen crystal by a fraction of a degree resulted in a redistribution of diffracted intensity as may be seen by comparing topographs shown in Figs. 1b and 1c. The twin related domains within regions such as A were oriented for diffraction in Fig. 1b but not in Fig. 1c while the reverse was true for the domains in regions such as F. All regions of the crystal could be brought into a diffraction condition by varying the orientation of the crystal. The observed contrast cannot be caused by surface irregularities or topology as it is extremely sensitive to the crystal orientation. The contrast and its dependence on the orientation of the crystal was consistent with the crystallography of the twin related domains (Schober and Wenzl 1976) which form on hydrogen ordering from the α' solid solution to form the β hydride phase. Minor differences between the optical micro-graphs and the topographs were due to the depth of penetration of the x-rays and the resulting observation of the domain structures beneath the surface of the crystal.

An example of the domain structure in a hydride crystal of composition $NbH_{0.78}$ is given in Fig. 2. This topograph, taken with Mo K_α radiation, diffraction vector $\underline{g}$ = [220] and an L4 emulsion shows finer details than are visible in the topographs recorded on the DF-57 emulsions. Optical microscopy with oblique lighting and with polarized illumination was unsuccesssful in revealing the domain structure due to relatively poor surface preparation. Nevertheless, the absorption depth of the Mo K_α radiation was sufficiently large to minimize the effect of surface damage and allow the domains to be imaged.

Two types of boundaries between neighboring domains were evident in the topographs of the $NbH_{0.78}$ and $NbD_{0.75}$ crystals. As discussed by Schober and Linke (1976b), the straight boundaries correspond to coherent twin boundaries between hydride (deuteride) domains, and the irregular boundaries correspond to incoherent twin boundaries. Trace analysis of the coherent boundaries revealed by x-ray topography is consistent with the interface planes identified by Schober and Linke (1976b): $\{100\}_c$, $\{110\}_c$, $\{111\}_c$, $\{210\}_c$ and $\{211\}_c$.

As indicated by observations similar to those discussed above, x-ray diffraction topography provides a practical alternative to optical metallography for the study of hydride domain structures. This technique has the advantages of requiring minimal surface preparation and of being a relatively rapid procedure.

ACKNOWLEDGEMENTS

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REFERENCES

Birnbaum, H.K., Grossbeck, M.L. and Amano, M. (1976). J. Less-Common Met., 49, 357-370.

Boettinger, W.J., Burdette, H.E., Kwiyana, M. and Green, R.E., Jr. (1976). Rev. Sci. Instrum., 47, 906-911.

Schober, T. (1975). Phys. Stat. Sol. (a), 29, 395-406.

Schober, T. and Linke, U. (1976a). Metallography, 9, 309-320.

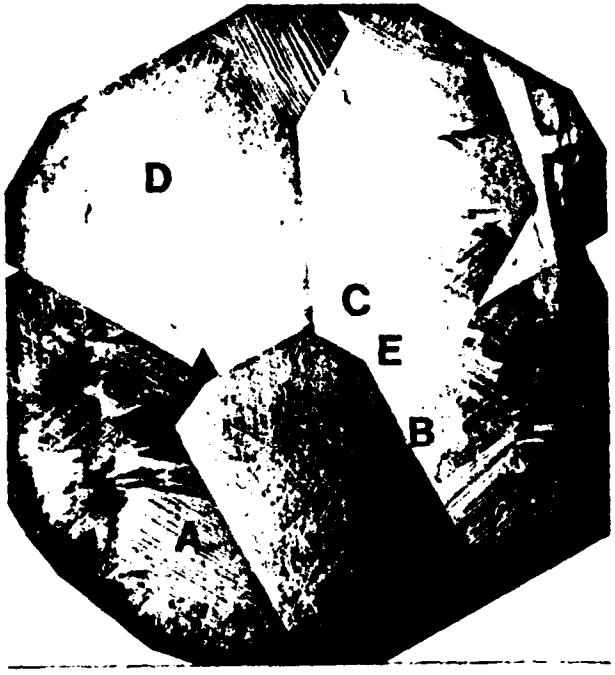
Schober, T. and Linke, U. (1976b). J. Less-Common Met., 49, 63-86.

Schober, T. and Wenzl, H. (1976). Phys. Stat. Sol. (a), 33, 673-681.

FIGURE CAPTIONS

Figure 1. Optical micrograph and corresponding ACT x-ray topographs showing the domain structure in a $NbD_{0.75}$ crystal. Fig. 1a is the optical micrograph taken with oblique illumination. Figs. 1b and 1c are x-ray topographs with the crystal in different orientations to bring different domains into a diffracting orientation. The images were recorded using Cu K_α radiation and $\underline{g}$ = [220] on double sided Kodak DF-57 film. The double sided film resulted in a loss of resolution but allowed images to be recorded in 45 mintues. The magnification for all images is as indicated on Fig. 1c.

Figure 2. ACT topograph of a $NbH_{0.78}$ crystal taken with Mo K_α radiation and $\underline{g}$ = [220]. The image was recorded on Ilford L4, 50 μm thick emulsion which improved the resolution as compared to the results obtained with the double sided film (Figs. 1b and c).



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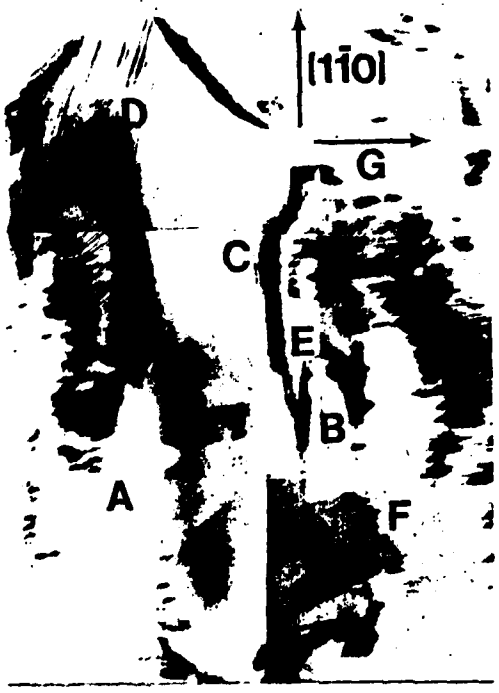

b

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Fig. 1
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Fig 2
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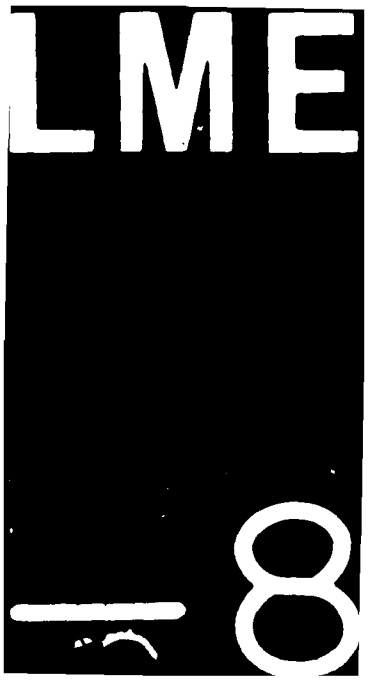
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