CONF. 790653-9

DIRECT OBSERVATION OF DEFECTS IN Al₅ COMPOUNDS
PRODUCED BY FAST NEUTRON IRRADIATIONS

C. S. Pande
Brookhaven National Laboratory
Upton, New York

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

MASTER

ALL INFORMATION CONTAINED HEREIN IS UNCLASSIFIED

28

By acceptance of this article, the publisher and/or recipient acknowledges the U.S. Government's right to retain a nonexclusive, royalty-free license in and to any copyright covering this paper.

DIRECT OBSERVATION OF DEFECTS IN A15 COMPOUNDS
PRODUCED BY FAST NEUTRON IRRADIATION¹

C. S. Pande
Brookhaven National Laboratory
Upton, New York

The nature of defect or defect complexes produced in superconducting compounds Nb₃Sn, Nb₃Pt, and V₃Si by high energy ($E \geq 1$ MeV) neutron irradiation is investigated by transmission electron microscopy. The newly developed technique of superlattice reflection imaging is used whereby the regions of reduced long range order are directly imaged. Unlike metals these regions were found in general not to collapse into dislocation loops. The size and the volume fraction of these disordered regions are obtained for fluences ranging from 10^{17} neutrons/cm² to 3×10^{19} neutrons/cm². The size ranges from 20 Å to 60 Å. Typical volume fraction for 10^{18} neutrons/cm² is over 1%.

I. INTRODUCTION

Marwick (1975) has estimated that more than 95% of the displacements produced in a metal undergoing neutron irradiation in a fast reactor occur within displacement cascades. In intermetallic compounds like A15s the cascades will also contain replacements (i.e. antisite defects). (In addition there is a possibility of the creation of antisite defects by the operation of long range collision sequences.) These cascades or disordered regions were extensively observed in Nb₃Sn using transmission electron microscopy (Pande, 1979), employing the newly developed superlattice reflection technique (Jenkins et al., 1976). For sufficiently low fluences, the specimens contain volumes of cascades where the long range order S is

¹This work was performed under the auspices of the U.S. Department of Energy.

much smaller than the surrounding matrix. In the superlattice reflection technique regions of low S are mapped as dark dots. The contrast arises due to the difference in structure factor between the disordered zones and the more or less ordered matrix. So far the evidence for the disordered regions were obtained only in Nb_3Sn , by transmission electron microscopy (Pande 1977, 1979) and low angle scattering (Karkin et al., 1976). It is of interest to know whether other A15 compounds such as Nb_3Pt , V_3Si show similar behavior. The chief aims of the present work are:

- 1) to use the superlattice reflection technique to investigate irradiated Nb_3Sn , Nb_3Pt , and V_3Si ,
- 2) to estimate the volume fraction of the disordered regions in Nb_3Sn and to compare with what would be expected theoretically, if all antisite defects are confined to these disordered regions.

II. SPECIMEN PREPARATION

Nb_3Sn specimens were prepared by solid state diffusion ("bronze") process. The grain size of such a sample is usually of the order of μ and is suitable for transmission electron microscopy in the diffraction contrast modes, although detailed Burgers vector determination is very difficult. V_3Si and Nb_3Pt were prepared by arc melting. Both of them had a grain size of over 1μ .

Discs of these materials ~ 3 mm in diameter are sealed in quartz tubing. The quartz tube is then placed in an aluminum container and surrounded with fine mesh aluminum powder and held in place with aluminum foil. The irradiation was carried out in the Brookhaven High Flux Beam Reactor (HFBR). The specimen temperature during the irradiation is expected to be $150^\circ\text{--}250^\circ\text{C}$. The fluences mentioned in this paper refer only to neutrons with energy $E \geq 1$ MeV, i.e. high energy neutrons with a flux of 1.3×10^{14} n/cm²-sec. However neutrons of lower energy as well as thermal neutrons are also present.

For obtaining specimens suitable for transmission electron microscopy, ion thinning procedure was used. The iron milling was done at a grazing angle of $12\text{--}15^\circ$ and Argon ions were accelerated with voltage of 4.5-5 kV. Ion current was 25-50 microamperes and gun current ranging from 0.4 to 2 milli-amperes. In some cases the specimens were washed in HF to remove damage layers due to ion milling.

III. EXPERIMENTAL OBSERVATIONS AND RESULTS

The irradiated specimens were examined using diffraction contrast technique employing both fundamental and superlattice reflections. In Nb_3Sn and Nb_3Pt 'small black dots' (disordered regions) were observed in specimens irradiated to various fluences. For example Fig. 1 shows a single Nb_3Sn grain of the irradiated specimen imaged in superlattice reflection ($g=011$) showing a high density of 'black dots'. As stated previously we identify the 'black dots' in dark field superlattice reflection as arising from the disordered regions created by the high energy neutrons and that the image is a simple projection of the disordered regions on the imaging plane [(100) in this case]. The size of the contrast figures under these conditions was taken to be the size of the disordered regions as they are close to spherical shape, although some of the disordered regions appear to be slightly elliptical. Figure 2 shows similar disordered regions in Nb_3Pt .

In the case of V_3Si however this technique has so far not been successful. We have not observed disordered regions in V_3Si on neutron irradiation after repeated attempts. This result is in agreement with that of Küpfer and Manuel (1979) and probably indicates annealing of defects in V_3Si at the reactor temperature. Unlike metals, very few of the disordered regions were found to collapse into dislocation loops.

The volume F_V of the disordered regions is an important parameter in calculating T_C degradation of the material on irradiation. This was done by measuring the disordered regions and measuring the thickness of the foil. It should be emphasized that F_V so obtained are rough estimates only. Figure 3 is a plot of the volume fraction of the disordered regions as a function of fluence for irradiated Nb_3Sn . It is clear that F_V is not insignificant as sometimes claimed. The theoretical curve is obtained on the assumption that all the antisite defects are present in the disordered regions. Experimental value of k as obtained by Sweedler and Cox, 1975 is used to estimate theoretical F_V (see Pande (1977) for details).

IV. CONCLUSIONS

1) Disordered regions in two A15 compounds, viz Nb_3Sn , and Nb_3Pt , produced by fission neutron irradiation at reactor temperature are directly imaged.

2) Volume fraction of the disordered regions is measured from the electron micrographs. It is shown that the majority of defects in Nb_3Sn are present in these disordered regions.

3) No disordered regions were observed in V_3Si irradiated at reactor temperature indicating the possibility of annealing of the defects at this temperature.

ACKNOWLEDGMENTS

The author is grateful to Dr. M. Suenaga for initiating this work and for many valuable suggestions and comments. Superb technical assistance by Messers. Bob Jones and Frank Thomsen is also gratefully acknowledged.

REFERENCES

- Jenkins, M. L., Katerbau, K. H., and Wilkens, M. (1976). Phil. Mag. 34, 1141.
- Karkin, A. E., Arkhipov, V. E., Ghoshchitskii, B. N., Romanov, E. P., and Sidorov, S. K. (1976). Phys. Stat. Sol. (a) 38, 433.
- Küpfer, H. and Manuel, A. A. (1979). Phys. Stat. Sol. (a) (in press).
- Marwick, A. D. (1975). J. Nucl. Mater. 55, 259.
- Pande, C. S. (1979). Phys. Stat. Sol. (a) 52, 687.
- Pande, C. S. (1977). Solid State Commun. 24, 241.
- Sweedler, A. R. and Cox, D. E. (1975). Phys. Rev. B12, 147.

FIGURE 1. *Disordered regions (black dots) in irradiated Nb₃Sn. (Fluence $\sim 10^{19}$ neutrons/cm²).*

FIGURE 2. *Disordered regions in irradiated Nb₃Pt. (Fluence $\leq 10^{17}$ neutrons/cm².) Diffraction conditions are shown on bottom right hand corner. Top left hand corner shows a magnified view of one of the disordered regions.*

FIGURE 3. *Volume fraction F_v of the disordered regions as a function of fluence in Nb₃Sn. (See text for details.)*