X-ray Diffraction Investigation of Single Crystal Niobium

R. K. Bollinger\textsuperscript{1}, Y. Suzuki\textsuperscript{2}, B. White\textsuperscript{1}, C. M. dos Santos\textsuperscript{3}, H. R. Z. Sandim\textsuperscript{3}, and J. J. Neumeier\textsuperscript{1}, S. Francoual\textsuperscript{4}, M. von Zimmerman\textsuperscript{4}

\textsuperscript{1}Department of Physics, Montana State University, P. O. Box 173840, Bozeman, Montana 59717-3841, USA, 
\textsuperscript{2}National High Magnetic Field Laboratory - Los Alamos National Laboratory, Los Alamos, New Mexico, 87545, USA 
\textsuperscript{3}Escola de Engenharia de Lorena - Universidade de São Paulo, P. O. Box 116, Lorena-SP, 12602-810, Brazil, 
\textsuperscript{4}HASYLAB at DESY, Notkestr. 85, D-22607 HAMBURG, Germany.

A number of anomalous physical properties have been observed in high-purity Niobium.[1] These anomalies occur at high temperature (150 K < T < 300 K), and include anisotropic thermal expansion,[2] as determined by a fused-silica capacitive dilatometer.[3] The thermal expansion results strongly suggest a potential lowering of symmetry, although the lower symmetry structure and the fraction of the material that transforms cannot be determined from thermal expansion alone. An X-ray study was thus necessary to explore the nature of the apparent phase transition. Informing the experimental approach were numerous reports of similar physical properties in the A15 superconductors, V\textsubscript{3}Si and Nb\textsubscript{3}Sn. These materials are known to undergo a Martensitic phase transition at low temperature (just above the superconducting transition temperature), becoming tetragonal, while the high temperature parent phase is cubic.[4] In addition, some of the A15’s are known to not transform if they are not pure. This is also the case for Nb, as samples that have been heat-treated for strain relief no longer show any evidence of the transition. The residual resistivity ratio of these samples is considerably lower than for the virgin samples, indicating the uptake of impurities. An X-ray study on a non-transforming sample of single crystal Nb was previously reported.[2]

X-ray diffraction on a transforming sample was carried out at BW5 high energy beamline equipped with a triple crystal diffractometer at 110 keV, at which energy the attenuation length for Nb is 1.43 mm. The high-resolution set-up was used to look for peak-splitting of the (200), (020), and (002) Bragg reflections, as observed in the A15’s.[4] The sample was mounted in a displex cryostat to allow experiments to be performed between 60 K and 300 K, a range that includes the entire range of the observed anisotropy in thermal expansion. Fitting was more robust with two peaks at the lower temperatures studied (see Fig. 1). However, the splitting is an order of magnitude too small to explain the observed anisotropy in thermal expansion and the derived lattice parameters from the three orthogonal directions were the same to within the experimental error for any given temperature. However, as can be seen in Fig. 2, there was significant, and anisotropic, broadening of the Bragg peaks at lower temperatures in the transforming sample. The width of the Bragg peaks in the non-transforming sample are also plotted. They show no temperature dependent broadening or anisotropy and are resolution limited.

In Fig. 2, an hk mesh is plotted for Nb at 60 K. The data shown are a fraction of the data collected (h was scanned from 1.97 to 2.03 and k was scanned from -0.03 to 0.03). No satellite peaks were discovered. However, as it was assumed that peak-splitting would be observed, only a small fraction of reciprocal space was explored.

In the A15 compounds, forbidden Bragg peaks occur at temperatures well above the martensitic phase transition temperature.[4] Broadening of the Bragg peaks has also been observed with X-ray diffraction in the A15’s. Time constraints did not allow for the exploration of forbidden Bragg peaks in Nb at BW5 in this experimental run. However, despite the absence of peak splitting at low temperature in Nb, there remain significant similarities between elemental Nb and the A15 superconductors, and it is probable that forbidden peaks will be discovered in transforming Nb.
Figure 1: Left: Half-width half maximum of the $<200>$ Bragg peaks as a function of temperature for transforming (as received) and non-transforming (heat-treated) Nb samples. The jump at low temperature is due to the data being better fit with two peaks. Right: Broadening and reduction of intensity with decreasing temperature of the (200) Bragg peak.

Figure 2: An hk mesh in the vicinity of the (200) Bragg peak at 60 K in Nb. No satellite peaks were observed.

Thus, in the next run, a low resolution survey of hkl space will be desirable to search for forbidden Bragg peaks, and put to the test the proposition that the proposed martensitic phase transition in pure Nb is similar to that reported for the A15 superconductors. Preliminary work at Montana State University will be done to attempt to narrow the focus of the search.

References