Textures and Microstructures, 1990, Vol. 13, pp. 55–58
Reprints available directly from the publisher.
Photocopying permitted by license only

© 1990 Gordon and Breach Science Publishers S.A.
Printed in the United Kingdom

TEXTURE INVESTIGATIONS AT LOW TEMPERATURES BY NEUTRON DIFFRACTION SHOWN ON THE EXAMPLE OF TbAg

F. ELF, W. SCHÄFER, S. HÖFLER and G. WILL

Mineralogical Institute, University Bonn, Poppelsdorfer Schloß, D-5300 Bonn 1, West Germany

(Received November 11, 1989; in final form May 12, 1990)

The first low temperature texture investigation by neutron diffraction is presented. Pole figures are collected on a special full circle Eulerian cradle mounted completely inside a helium cryostat. Nuclear and magnetic pole figures of antiferromagnetic TbAg are measured at 300 and at 77 K respectively. The bulk specimen revealed only a weak texture. Magnetic satellite reflections presumed for special sample orientations were not observed.

KEY WORDS Neutron diffraction, low temperature 77 K, pole figure measurement, magnetic reflexious, TbAg.

INTRODUCTION

There are cases, like the one demonstrated here, where the texture of a sample is needed at low temperature. Materials to be used in space applications require such studies, samples which undergo a magnetic phase transformation and where a different texture after the phase transformation, normally accompanied by a crystallographic transformation may be expected. The memory metals would be another example.

Neutron diffraction is especially well suited to face such problems because of the high penetration power, e.g. low absorption of neutrons in material (Will, Schäfer and Merz, 1989). The neutron windows (generally aluminium) of the cryostat are easily transmitted by the neutrons. The general advantage of neutron diffraction, large samples for example, is another specific point of interest.

We report here a texture investigation by neutron diffraction for the first time on a sample at 77 K in a cryostat. The sample is TbAg, which crystallizes at room temperature in the CsCI-type structure (space group $Pm\bar{3}m$) (Cable, Koehler and Wollan, 1964). On cooling below the magnetic phase transformation at $T_N = 100\,K$ the Tb sublattice experiences collinear antiferromagnetic long range ordering. The tetragonal unit cell of the magnetic structure follows from the cubic unit cell of the crystal structure by doubling the lattice parameter a_0. We have chosen TbAg, because in the analysis of the magnetic structure difficulties were experienced because of texture effects in the bulk sample.

EXPERIMENTAL

The measurements were done with the sample in a newly developed Eulerian cradle mounted inside a cryostat (Elf, Will, Chatzipetros and Dujka, 1984; Chatzipetros, Dujka, Elf and Will, 1987). Conventional Eulerian cradles for low temperature measurements are constructed in such a way that the ϕ-circle is off center on the χ-circle. The sample on the ϕ-circle is then replaced by the cryostate with the sample inside. This construction is rather easy and straight forward, however, it is handicaped by a limited range of ϕ- and χ-angles and thus difficult to use for texture analysis.

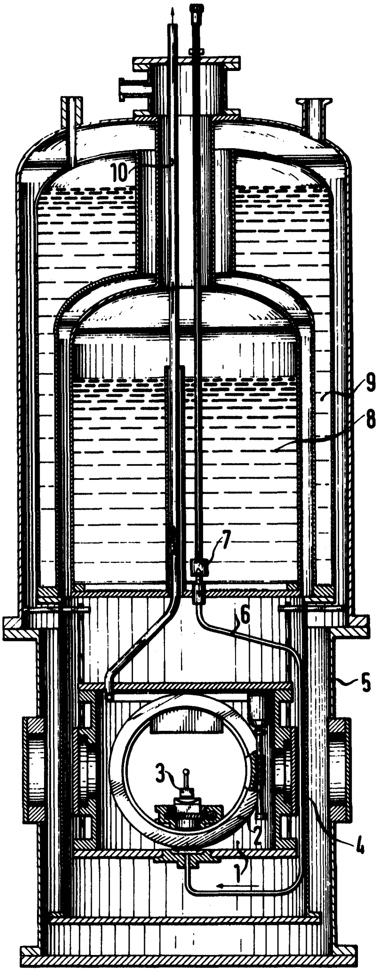

Figure 1 Schematic representation of the low temperature device with the Eulerian cradle inside the helium cryostat: (1) chamber for Eulerian cradle, (2) Eulerian cradle (χ-circle), (3) goniometer head, (4) radiation shield, (5) vacuum shield, (6) capillary tube for helium transfer to the sample chamber, (7) needle valve for temperature regulation, (8) liquid helium reservoir, (9) liquid nitrogen reservoir, (10) helium exhaust.

We have constructed a system, where the Eulerian cradle, ϕ- and χ-circles, are mounted completely inside a cryostate, including the stepping motors. The coolant, liquid helium or nitrogen, may reach the sample itself. The temperature is variable from 4.2 K (liquid helium) to room temperature, whereby the sample in these cases is cooled by helium gas. Figure 1 gives a picture of the apparatus.

The bulk TbAg sample was investigated at 300 and 77 K. The specimen had the form of an elliptical (7 mm by 5 mm) column 10 mm high. Geometrical absorption effects could be neglected.

RESULTS

We have measured pole figures 110 at room temperature and at 77 K, and also the pole figure of the reflection $\frac{1}{2}\frac{1}{2}0$ at 77 K. These three pole figures are depicted in Figure 2 in stereographic projection. All pole figures have been measured in an approached "equal area scanning" mode: The increments in azimuth $\Delta\phi$ are 5° for the outer parts ($0° \leq \chi \leq 25°$), 10° for $30° \leq \chi \leq 55°$ and 20° for $60° \leq \chi \leq 90°$. Increments in pole distance $\Delta\chi$ are 5°, giving a total of 757 data points on the hemisphere. The measuring time was about 15 hours per pole figure.

The 110 pole figures at 300 K and 77 K are nearly identical, exhibiting a small preferred orientation of the (110) crystallographic planes. The orientation is parallel to the cylinder axis of the sample. The preferred orientation effect is weakly reduced for the corresponding magnetic superstructure reflection.

This investigation was attempted because we expected additional satellite reflections $\frac{1}{2}\frac{1}{2}1^{(-)}$ and $\frac{1}{2}\frac{1}{2}1^{(+)}$ at the left and right side of the $\frac{1}{2}\frac{1}{2}1$ magnetic reflection. Such satellites have been found in the magnetically diluted compounds $\mathrm{Tb}_x\mathrm{Y}_{1-x}\mathrm{Ag}$ (Maletta and Schäfer, 1986). For this reason we measured a second set of pole figures at 77 K: $\frac{1}{2}\frac{1}{2}1^{(-)}$ and $\frac{1}{2}\frac{1}{2}1^{(+)}$. They are shown in Figure 3 together with the central of position $\frac{1}{2}\frac{1}{2}1$. The satellites are with $\Delta 2\theta = \pm 0.2°$ very close to the $\frac{1}{2}\frac{1}{2}1$ position, so they would show up at the tails of such a reflection. These satellite positions would correspond to a propagation vector of an incommensurate magnetic component along the c-axis with a periodicity of about 58 Å. As can be seen from Figure 3 the three pole figures are basicly identical and therefore we have to conclude that there are no satellite reflections in the sample. Small differences between $\frac{1}{2}\frac{1}{2}1^{(-)}$ and $\frac{1}{2}\frac{1}{2}1^{(+)}$ are due to experimental uncertainties.

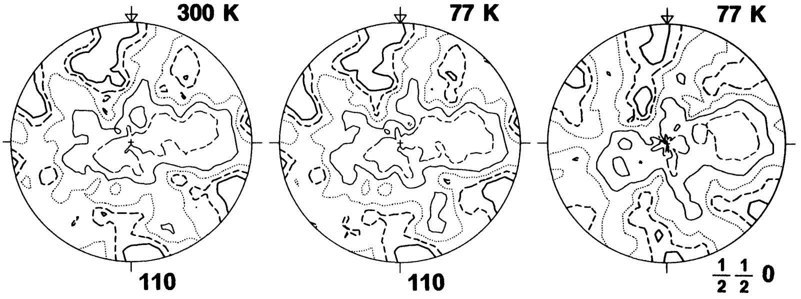

Figure 2 Pole figures of TbAg. 110 at room temperature (left), 110 at 77 K (middle), $\frac{1}{2}\frac{1}{2}0$ at 77 K (right). Maximum and minimum pole densities are 2.29 and 0.17 (left), 2.37 and 0.19 (middle), 1.94 and 0.24 (right) respectively. The arrows indicate the direction of the primary neutron beam.

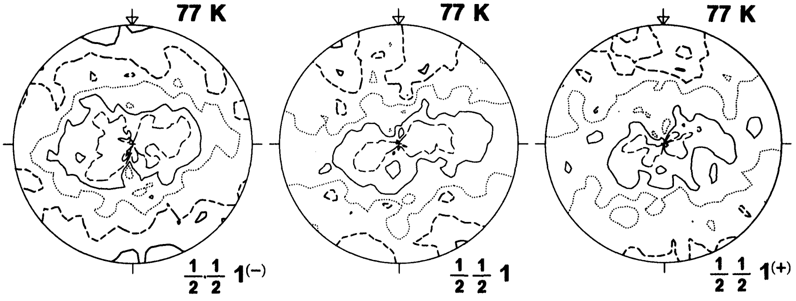

Figure 3 Pole figures of TbAg. Presumed left satellite $\frac{1}{2}\frac{1}{2}1^{(-)}$ (left), central magnetic reflection $\frac{1}{2}\frac{1}{2}1$ (middle), presumed right satellite $\frac{1}{2}\frac{1}{2}1^{(+)}$ (right), all at 77 K. Maximum and minimum pole densities are 1.98 and 0.04 (left), 1.74 and 0.09 (middle), 1.72 and 0.14 (right) respectively. The arrows indicate the direction of the primary neutron beam.

DISCUSSION AND CONCLUSION

This example demonstrates the big advantage of neutron diffraction in texture analysis when specific requirements are needed: for example low temperature and in this specific case and also very important the investigation of magnetic behavior. We could measure complete pole figures over all ϕ- and χ-angles without limitations. The big advantage of our specific low temperature device in comparison with conventional Eulerian cradles with a small cryostate at the sample position is also found that it is not necessary to correct for absorption as a function of angle dependent window transmission.

Acknowlegdments

We wish to thank Dr. H. Maletta, Institut für Festkörperforschung, KFA Jülich, for providing the sample. This work has been funded by the German Federal Minister of Research and Technology (BMFT) under contract number 03WI1BON.

References

Cable, J. W., Koehler, W. C. and Wollan, E. O. (1964). Magnetic Order in Rare-Earth Intermetallic Compounds. *Phys. Rev.* **136,** A240–242.

Chatzipetros, J., Dujka, B., Elf, F. and Will, G. (1987). Low-temperature goniometer for X-ray and neutron diffractometry. United States Patent 4,644,761.

Elf, F., Will, G., Chatzipetros, J. and Dujka, B. (1984). Full-circle Eulerian cradle for low temperature neutron investigation. *Revue Phys. Appl.* **19,** 793–794.

Maletta, H. and Schäfer, W. (1986). Änderung der antiferromagnetischen Ordnung von TbAg durch Verdünnung in $Tb_xY_{1-x}Ag$. *Verhandlungen der Deutschen Physikalischen Gesellschaft* **5,** 1202.

Will, G., Schäfer, W. and Merz, P. (1989). Texture Analysis by Neutron Diffraction Using a Linear Position Sensitive Dector. *Textures and Microstructures* **10,** 375–387.