

representative of bulk properties appears to have been eliminated by the independence of these results with respect to particle size (as long as the particle sizes are approximately less than the microwave skin depth).

These experiments are now being extended to other temperatures and a study of a number of calibration standards is being carried out which, it is hoped, will allow accurate absolute susceptibility measurements.

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## X-Ray Topographic Observation of Antiferromagnetic Domain Boundaries in Chromium

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Antiferromagnetic domains in Cr have been observed visually by the x-ray double-crystal method in the low-temperature phase which corresponds to the longitudinal spin-density-wave state. The contrast between neighboring domains is due to a slight tetragonality of the crystal lattice. The domain boundary has been found to be sharp and mobile, and its section is straight if not trapped by imperfections.

There have been several x-ray topographic investigations on antiferromagnetic domains. The so-called *T* walls were easily detected in NiO<sup>1,2</sup> and CoO,<sup>3</sup> because of a relatively large rhombohedral distortion of the crystal lattice in each case. In NiO, the *S* walls were also detected<sup>4</sup> because of local strain of about  $10^{-4}$ . In the case of chromium, possible deviation from cubic symmetry in its antiferromagnetic state below the Néel temperature, 311°K, has not been detected by the x-ray-diffraction method,<sup>5,6</sup> at least in the temperature range surveyed. Recently this deviation was clearly shown by measurements using the strain-gauge technique<sup>7</sup> or the three-ter-

minal capacitance technique.<sup>8</sup> Although these measurements were carried out mainly in the higher temperature region, 122–311°K, where the crystal is in a transverse spin-density-wave state ( $AF_1$ ), the result obtained<sup>8</sup> suggested that the deviation  $(a-c)/a$  is of the order of  $10^{-5}$  in the temperature region lower than the spin-flip temperature, 122°K, where the crystal is in a state of longitudinal polarization ( $AF_2$ ).

In the present study, an attempt has been made to take an x-ray topograph of antiferromagnetic domain boundaries in the  $AF_2$  state by the method of double crystals in a parallel position using the 211 reflection of Cr, as shown in Fig. 1. Be-

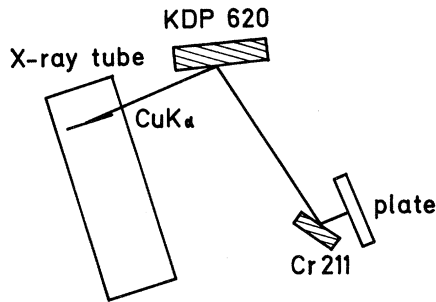


FIG. 1. Experimental arrangement. The Cr sample is kept in a vacuum chamber cooled by a cryostat.

cause a large, nearly perfect Cr crystal was not available, a nearly perfect potassium dihydrogen phosphate crystal was used as the first crystal because the interplanar spacing for 620 is different only by 0.094% from that for 211 of Cr. The sample,<sup>9</sup> with as-grown surfaces parallel to  $\{111\}$ , was used for topography after being electropolished in concentrated oxalic acid.

The topographs obtained at room temperature did not contain any indication of domain boundaries, as is shown in Fig. 2. This fact is reasonable because in this temperature region the deviation from cubic symmetry is very small according to the above-mentioned studies.<sup>7,8</sup> In contrast to this, the topograph at liquid-nitrogen temperature showed a boundary with black and white contrast on its two sides, as shown in Fig. 3. This contrast was reversed when the topograph was taken at the opposite side of the rocking curve, as is also seen in Fig. 3. Although a splitting of the peak profile corresponding to the different values of  $a$  and  $c$  was not found, it was estimated that the half-width of the rocking curve increased by about  $4''$  of arc in this spin state compared with its value at room temperature.

When the sample was left at room temperature,

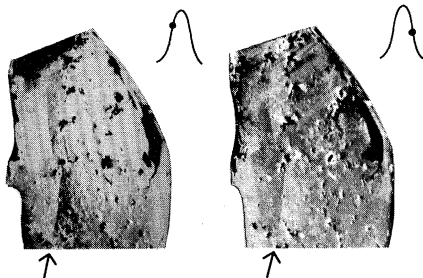


FIG. 3. Topographs taken at liquid-nitrogen temperature. The position of a domain boundary is shown by an arrow in each photo.

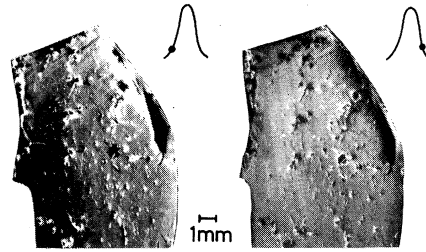


FIG. 2. Topographs taken at room temperature. The solid circle on each rocking curve indicates the angular position where the corresponding topograph was taken. The white region corresponds to the part upon which more x rays fell.

294°K, a little below the Néel temperature, for a few days and then cooled again to the same low temperature as before, the boundary in the topograph slightly shifted to the left and became almost straight as shown in Fig. 4. This fact suggests that the boundary seen in Fig. 3 was curved as a result of being trapped by imperfections.

The boundary observed can be considered to be due to different directions of tetragonal axes of antiferromagnetic domains on its two sides for the following reasons: (1) The boundary is observed only in the low-temperature region. (2) The feature of a very sharp boundary with a strong contrast between its two sides cannot be explained as a result of haphazard strain due to cooling. (3) The intersection of the boundary with the crystal surface is very straight and nearly parallel to the crystallographic direction  $[01\bar{1}]$  if it is not influenced by any crystal imperfection. (4) It has been shown from neutron-diffraction measurements that a nearly perfect Cr crystal often includes a few or a single domain even without being cooled in a magnetic field. (5) The boundary is mobile as in the case of S walls in NiO.

It is expected that the antiferromagnetic domain configuration in Cr may be different in every

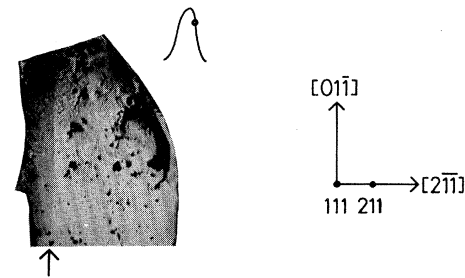


FIG. 4. A topograph taken with the sample re-cooled to low temperature after having been kept at room temperature for a few days.

crystal, being influenced by the stress field due to the existing imperfections. It seems to be already certain that the observed boundary has a magnetic origin. However, in order to get more direct proof, further experiments such as an attempt to find any field effect will be made.

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## Electric Quadrupole Coupling in $\alpha$ -Fe†

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The <sup>57</sup>Fe 14.4-keV-level electric-quadrupole coupling in  $\alpha$ -Fe has been detected by Mössbauer spectroscopy.

The purpose of this note is to point out that the <sup>57</sup>Fe 14.4-keV-level electric-quadrupole coupling in ferromagnetic  $\alpha$ -Fe is measurable by Mössbauer spectroscopy. A quadrupole effect was first observed in high-precision spectra of the National Bureau of Standards (NBS) Standard Reference Material (SRM) No. 1541,  $\alpha$ -Fe foil. Subsequently, 36 high-precision spectra from samples of this material have been measured on the NBS and Livermore absolute-velocity Mössbauer spectrometers at 298 and 4.3°K. These data confirm the presence of a quadrupole effect.

In previous studies<sup>1</sup> quadrupole coupling in  $\alpha$ -Fe was assumed to be zero because of cubic symmetry. However, even with cubic symmetry, a quadrupole effect can be magnetically induced.<sup>2</sup> Furthermore,  $\alpha$ -Fe is cubic only in the paramagnetic state. It must undergo a lattice distortion (magnetostriction) upon ferromagnetic ordering.<sup>3</sup> The magnetostriction of macroscopic  $\alpha$ -Fe samples indicates that the lattice distortion may be too small to be detected by standard x-ray techniques.

The material (SRM No. 1541) examined at both

laboratories is low-carbon, high-purity iron, rolled to a thickness of about 20  $\mu$ m. The impurities in weight percent are Ni, 0.03; Co, 0.02; Cr, 0.015; and Mn, 0.005. The carbon and oxygen contents are C, 60 ppm and O, 100 ppm. All other impurities are less than 0.001 wt%. The NBS absolute-velocity spectrometer uses an electromagnetic velocity transducer with an optical (Michelson) interferometer feedback loop, in the constant-acceleration mode.<sup>4</sup> The Livermore spectrometer is a mechanical constant-velocity system.<sup>5</sup> The velocity calibration of each spectrometer is derived from moiré-fringe distance measurements. The line positions were obtained by least-squares fitting, with no constraints, six Lorentzian lines to each spectrum (nineteen independent parameters). The  $\alpha$ -Fe quadrupole effect based on sixteen NBS runs with the sample at 298°K, and on twenty Livermore runs, twelve with the sample at 298°K and eight at 4.3°K, is given below. The 298°K results of the two laboratories are in excellent agreement and the weighted mean is quoted. The accuracies of both the NBS and Livermore results were determined by

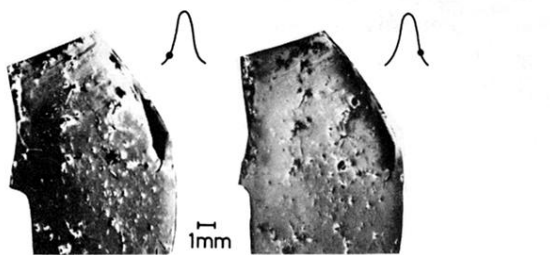


FIG. 2. Topographs taken at room temperature. The solid circle on each rocking curve indicates the angular position where the corresponding topograph was taken. The white region corresponds to the part upon which more x rays fell.

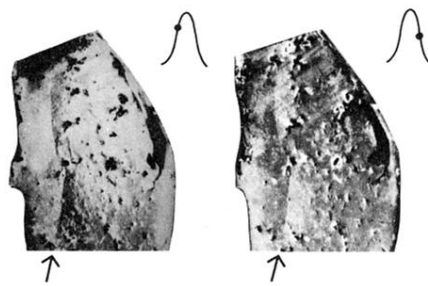


FIG. 3. Topographs taken at liquid-nitrogen temperature. The position of a domain boundary is shown by an arrow in each photo.

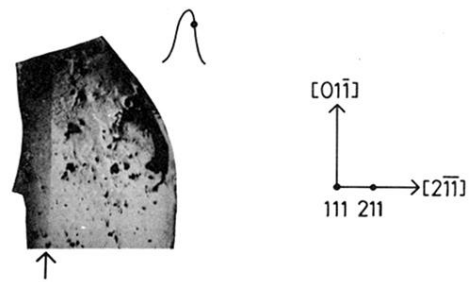


FIG. 4. A topograph taken with the sample recooled to low temperature after having been kept at room temperature for a few days.