JOURNAL OF SOLID STATE CHEMISTRY 48, 368–375 (1983)

High Resolution Electron-Microscope Studies on a Quenched $(Ni_{0.5}Cr_{0.5})_7Nb_6$ Alloy

L. DOUXING,* L. STENBERG,† AND S. ANDERSSON

Inorganic Chemistry 2, Chemical Center, P.O. Box 740, S-220 07 Lund, Sweden

Received December 15, 1982; in revised form March 18, 1983

Crystals of a $(Ni_{0.5}Cr_{0.5})_7Nb_6$ alloy have been studied with a high-resolution electron microscope. Crystals of the Friauf–Laves and μ phases were identified in the sample, which is in agreement with the X-ray powder pattern. Planar defects of different kinds were easily and frequently observed. The structural model of defects have been derived via chemical twinning and intergrowth operations.

Introduction

High-resolution electron microscopy has been very useful in the study of the real structure of crystals on an atomic scale. Two-dimensional lattice images recorded with a high-resolution electron microscope not only give the average structure, but also reveal the different kinds of structural defects. The high-resolution electron-microscopy technique has been applied to the study of some of the so-called tetrahedrally close-packed alloy structures. Two-dimensional lattice images of regular and defect structures were found for the μ phase in Fe–Mo and Mo–Co–Si alloys with a high-resolution electron microscope (*1, 2*). Different crystallographic operations and intergrowth have been used to describe the so-called tetrahedrally close-packed alloy structures (*3*). This concept will be used here since it is general and nearly exact in deriving crystal structures and their planar defects. The Mg-based pseudo-binary Friauf–Laves phases have been extensively investigated and several stacking variants and defects have been found by Y. Komura *et al.* (*4*).

The aim of the present investigation was to study the nature of the defect structures of the Friauf–Laves and μ phases as they occur in the $(Ni_{0.5}Cr_{0.5})_7Nb_6$ alloy with a high-resolution electron microscope.

Experimental Procedure

Alloys having the nominal composition $(Ni_{0.5}Cr_{0.5})_7Nb_6$ were prepared by melting weighed amounts of pure Nb, Ni, and Cr in an argon-filled electric arc furnace. The sample was sealed in a silica tube, which was evacuated and annealed at 1055°C for 24 hr. The powder pattern of the sample obtained in a Guinier–Hägg focusing camera using Cu$K\alpha$ radiation showed the presence of the Friauf–Laves and μ phases.

The crystals were crushed and ground in

* On leave from the Institute of Metal Research, Academia Sinica, Shenyang, China.

† To whom correspondence should be addressed.

0022-4596/83 $3.00

a hardened steel mortar and then thin fragments were collected on a holey carbon film supported on a copper grid. The perforated carbon films with the sample were transferred to a JEOL 200CX electron microscope, equipped with a top entry high-resolution $\pm 10°$ double tilting stage and operated at 200 kV. Crystals of the Friauf–Laves and μ phase were identified in the sample, which is in good agreement with the X-ray powder pattern. Using selected area diffraction and a goniometer stage, the orientation of the crystals were adjusted, so that the [110] of the hexagonal cell for Friauf–Laves and μ phase was parallel to the incident electron beam. The alignment procedure had to be repeated for different parts of the crystal, since a slight bending is always present. High-resolution lattice images were recorded at a magnification of 690,000, using an objective aperture corresponding to a radius of 0.34 $Å^{-1}$ in the diffraction pattern. The computer simulated images were obtained using the multislice method with a program written by M. A. O'Keefe.

Results

The $MgZn_2$-type Friauf–Laves Phase

The crystal structure of the Friauf–Laves phase was found by Friauf (*5*) and Laves (*6*). The atomic arrangement in the $MgZn_2$-type Friauf–Laves structure projected along [110] can be described as consisting of uncentered trigonal bipyramids sharing edges and corners generating pentagonal antiprisms. This can be seen in the inserted drawing in the high-resolution micrograph (Fig. 1) of a perfectly ordered structure of

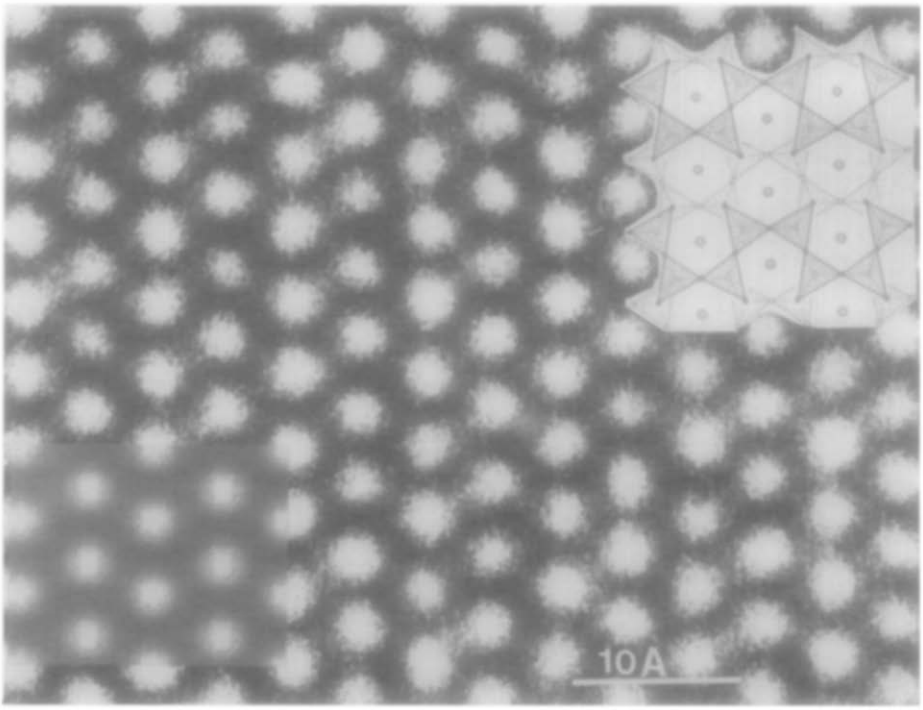

FIG. 1. Crystal structure image (CSI) of a perfectly ordered $(Ni,Cr)_7Nb_6$ crystal with $MgZn_2$ structure in the [110] direction. Crystal structure drawing and calculated image are inserted in upper right and lower left corners, respectively.

$MgZn_2$ type in the [110] direction. The white dots correspond to the position of the pentagonal antiprisms, which was confirmed by calculation with the multislice method; see insertion in lower left corner of Fig. 1. The following parameters were used for the calculation.

Spherical aberration coefficient = 1.2 mm, slice thickness = 4.861 Å, the focus spread due to chromatic aberration = 30 Å, incident beam convergence = 0.6 mrad, and the number of diffracted beams included in the dynamical diffraction calculation = 1027. The images were computed for a range −500 to −800 Å. The best agreement between experimental and calculated image was made with a defocus of −600 Å and a crystal thickness of 38.9 Å and this was used to produce the inserted image.

Planar defects of different kinds were easily and frequently observed. Figure 2 shows a two-dimensional crystal structure image of a well-resolved planar defect in $MgZn_2$-type Friauf–Laves phase structure. On the basis of the correlation of the image contrast with structural features, the structure of the defects can be derived from the observed image contrast. A structural model of the defect is inserted in Fig. 2. The structure of the defect can be described as an intergrowth between Friauf–Laves and three sheets of μ-phase-type structure (see below). Such an intergrowth structure has not yet been found in crystals of the Friauf–Laves phase structure. The crystal structure image in Fig. 3 shows another kind of defect, revealing an intergrowth between Friauf–Laves and four sheets of double-twinned μ-phase structural type. A model of the atomic arrangement of the defect area is shown in the figure, where the arrows show the twin-planes.

The μ Phase

The structure of the μ phase was first determined by Westgren (*7*) and was later refined for the isotypic Co_7Mo_6 (*8*). The μ-phase structure can be seen as an

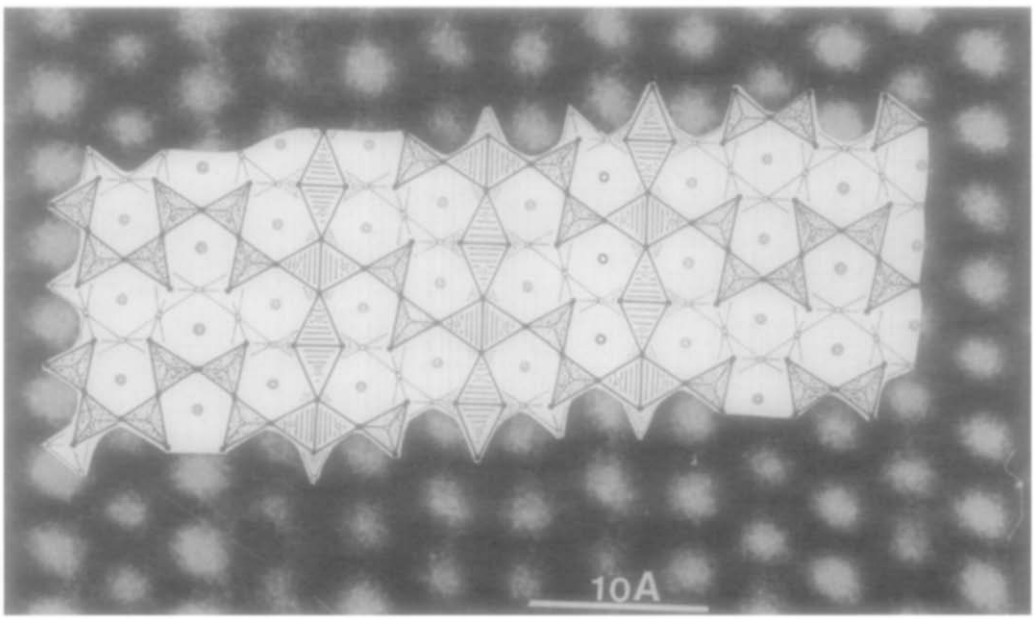

FIG. 2. $(Ni,Cr)_7Nb_6$ crystal with $MgZn_2$ structure, but with three slabs of the μ-phase structure penetrating the crystal.

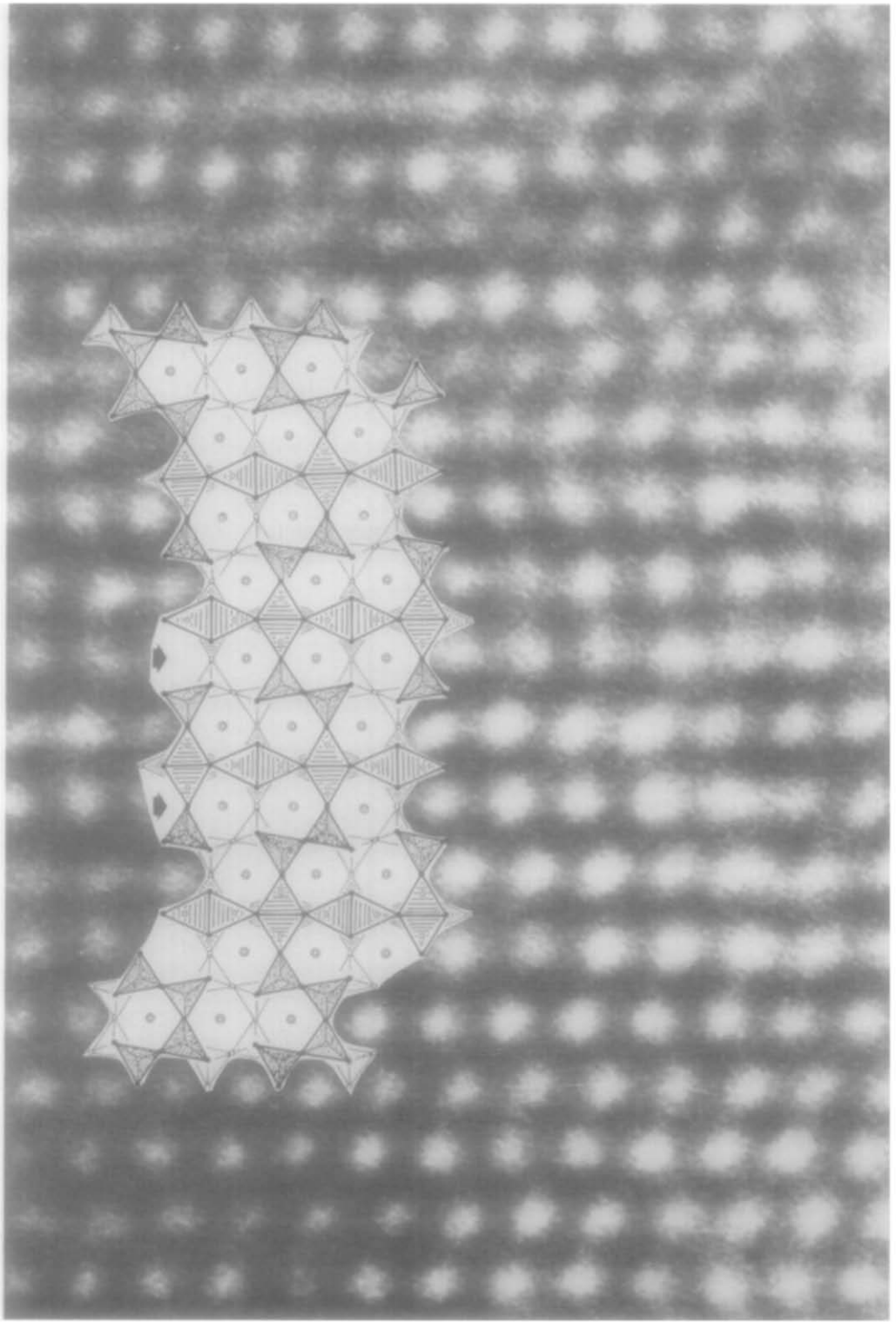

FIG. 3. CSI of $(Ni,Cr)_7Nb_6$ crystal with $MgZn_2$ structure with an intergrowth of twinned μ-phase structure or the Cs_7K_6 structure. Arrows indicate twin-planes.

intergrowth of sheets of the Zr_4Al_3, viewed along [1$\bar{1}$0], and $MgCu_2$ structure projected along [1$\bar{1}$0] (*3*). An interesting variation of the μ-phase structure has been found by A. Simon *et al.* (*9*) for the structure of Cs_6K_7.

The crystal structure image of a μ-phase crystal together with the structure drawing is shown in Fig. 4. The white dots indicate the sites of the pentagonal antiprisms generated by the structure. A calculated image, using the multislice method as mentioned above (also inserted in Fig. 4), agrees well with the experimental result.

In a previous paper (*2*) it was reported that the μ-phase structure could accommodate disorder in four simple ways, viz., twin operation and three kinds of intergrowth with a $MgCu_2$, $MgZn_2$, and Zr_4Al_3 structural type. The high-resolution image of the present observations has enabled us to obtain much more direct evidence of the presence and structure of defects of the μ phase. Figure 5 shows a crystal structure image of a twin defect with the atomic arrangement given in the inserted drawing. If a double twin operation is carried out in the two adjacent planes in [001] on the structure, the atomic arrangement of this type of defect can be achieved. Another way to describe this is obtained if the μ-phase structure were to intergrow with the Cs_6K_7 structural type. Another kind of well-resolved planar defect in a crystal of μ phase is shown in Fig. 6. The white dots in a region of a defect form a zigzag row revealing the presence of an intergrowth between the μ-phase structure and three sheets of the $MgZn_2$-type structure. The atomic arrange-

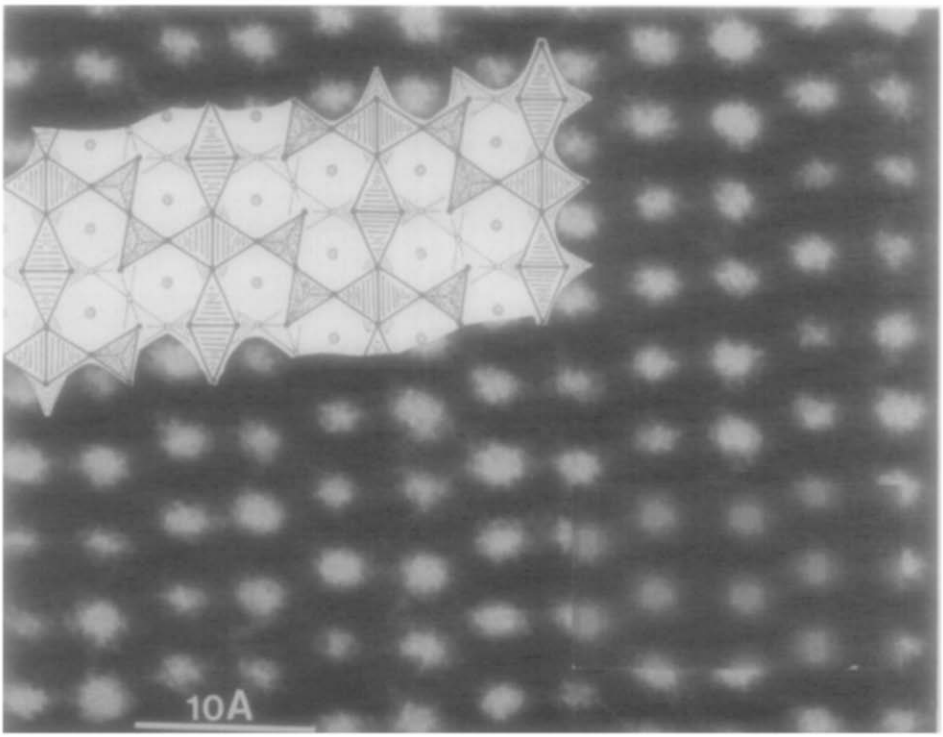

FIG. 4. CSI of a perfect $(Ni,Cr)_7Nb_6$ crystal with the μ-phase structure. The inserted calculated image was made with focus = −600 Å and thickness = 39.56 Å (8 slices).

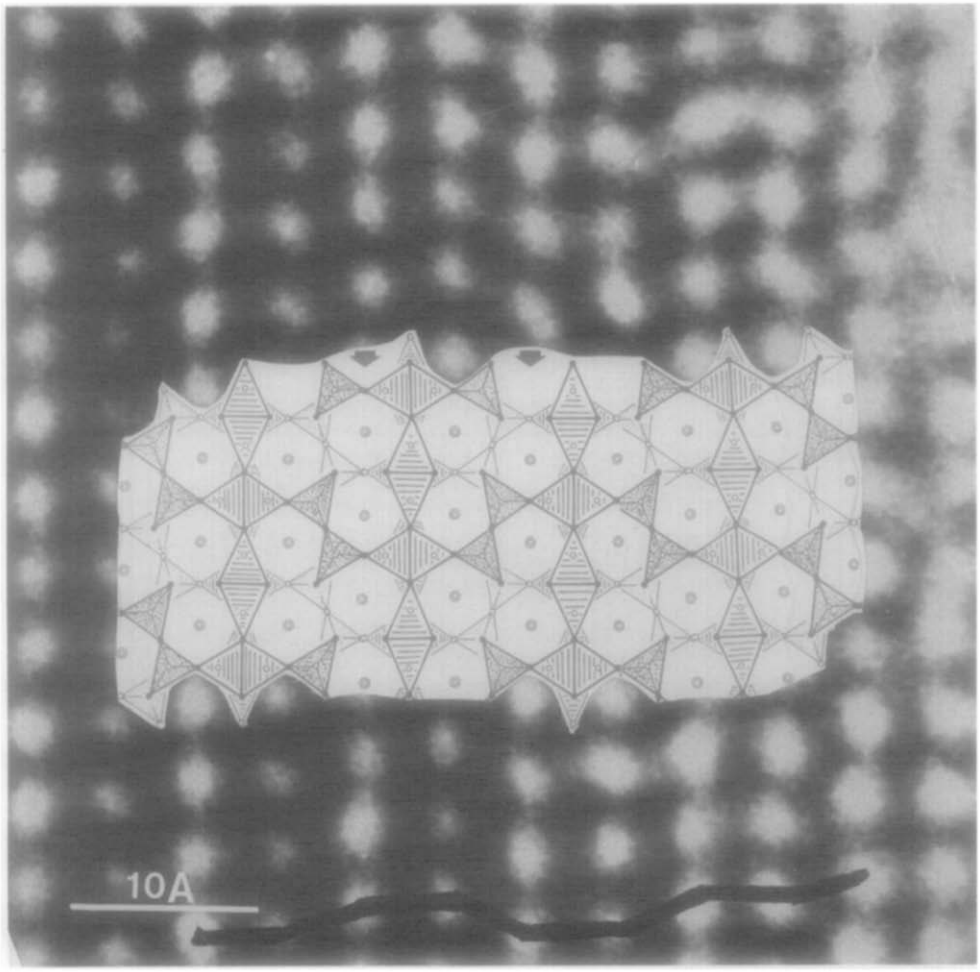

FIG. 5. Twin defects in a crystal with μ-phase structure. Arrows indicate twin-planes.

ment of the defect region derived from the image contrast is given in the inserted drawing.

Conclusions

It is clear that high-resolution electron microscopy is of great value for the investigation of the local defect structures of crystals of tetrahedrally close-packed alloys, which cannot be determined by other indirect methods. The Friauf–Laves and μ phases studied contain different kinds of defects. We have presented models for a variety of defect structures, which were derived by a direct correlation between contrast in a high-resolution lattice image and structural features. The structure of defects can be explained as derived from chemical twinning and intergrowth operations.

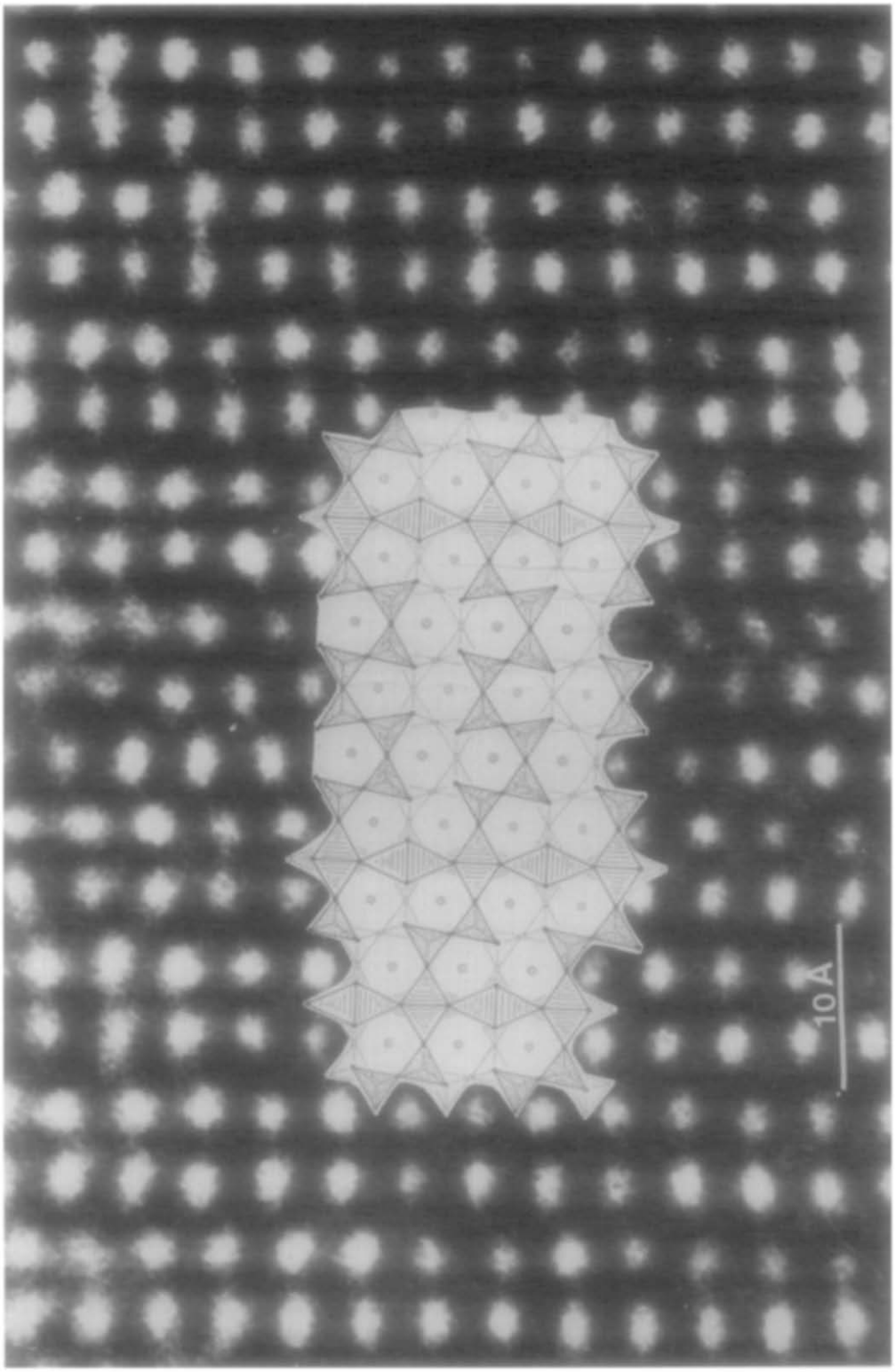

FIG. 6. Crystal with μ-phase structure intergrown with slabs with $MgZn_2$ structure.

Acknowledgments

This work was supported by the Swedish National Science Research Council. L. Douxing thanks the fellowship from the Royal Swedish Academy of Engineering Sciences.

References

1. L. STENBERG, *Chem. Scr.* **14,** 219 (1978–1979).
2. L. STENBERG AND S. ANDERSSON, *J. Solid State Chem.* **28,** 269 (1979).
3. S. ANDERSSON, *J. Solid State Chem.* **23,** 191 (1978).
4. Y. KITANO, Y. KOMURA, H. KAJIWARA, AND E. WATANABE, *Acta Crystallogr. Sect. A* **36,** 16 (1980).
5. J. B. FRIAUF, *Phys. Rev.* **29,** 34 (1927).
6. F. LAVES AND H. WITTE, *Metallwirt.* **15,** 840 (1936).
7. A. WESTGREN, Tokohu University, *Honda-Mem,* 852 (1936).
8. J. B. FORSYTH AND L. M. D'ALTE DA VEIGA, *Acta Crystallogr.* **15,** 543 (1962).
9. A. SIMON, W. BRÄMER, B. HILLENKÖTTER, AND H.-J. KULLMANN, *Z. Anorg. Allg. Chem.* **419,** 253 (1976).