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Decomposition and Ordering in $Fe_{1-x}O$

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Wüstite which exists above 570 °C, has a defect NaCl-type structure. The considerations of iron vacancies and interstitials depends on the deviation from stoichiometry. The defects are locally ordered; a tendency for long-range order is observed in quenched samples [Herai, T., Thomas, B., Manenc, J. & Benard, J. (1964). *C. R. Acad. Sci. Paris*, **258**, 4528–4530; Koch, F. & Cohen, J. B. (1969). *Acta Cryst.* B**25**, 275–287]. Samples in the composition range $Fe_{0.95}O$–$Fe_{0.88}O$ quenched from about 1000 °C, have been studied by electron diffraction/microscopy with special emphasis on the dark-field technique. Different quenching rates and compositions lead to a set of morphologies which can be described in terms of different stages of spinodal decomposition into an oxygen-rich and a near-stoichiometric component. The diffraction spots from the oxygen-rich component reveal a considerable degree of order, which is enhanced upon heat treatment at 300 °C, leading to a faulted superstructure. Previous diffraction studies have been interpreted in terms of an ordered phase with cubic symmetry. Dark-field microscopy, particularly at the symmetrical composition, shows that the compositional fluctuation wave contains 20 Å domains of tetragonal symmetry even at a very early stage. At asymmetrical compositions, and in the ordered component domains with tetragonal and orthorombic symmetry are found. Superstructure spots can be indexed by a *C*-face-centred orthorhombic unit cell with dimensions five times the fundamental unit cell. Reflexions satisfying the relation $h+5l=2n+1$ are extinguished. This rule and the intensities in the pattern indicate a periodic antiphase structure with period $M=2{\cdot}5$ and a shift displacement of half a fundamental unit cell.

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Antiphase Domains and Superlattice Spot Splitting in Cu_3Au I

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Lattice imaging by high-resolution electron microscopy has been utilized in the study of antiphase boundaries and antiphase domains in Cu_3Au I. A displacement of half the 100 fringe spacing characterizes the antiphase boundary and, by following the trace of 100 fringes from one ordered domain to another, the relative phase of adjacent domains may be established. The technique has shown that the discrete ordered domains formed in the early stages of ordering in Cu_3Au I are in small groups in which adjacent domains are in antiphase, with good correlation between the antiphase domain separation and the splitting of superlattice reflections in the corresponding diffraction patterns. It has also provided direct evidence that small regions of relatively high local degree of order (microdomains) exist in the disordered alloy.

Introduction

One of the principal features of diffraction patterns taken of Cu_3Au I alloys during the early stages of ordering is the splitting of the superlattice reflections. The character of the splitting is similar to, but not as marked as, that observed in Cu_3Au II, the long-period-superlattice form of the alloy in which antiphase boundaries (APB's) are present periodically throughout the lattice. The splitting has previously been interpreted in terms of a distribution of APB's in the material (Raether, 1952; Yamaguchi, Watanabe & Ogawa, 1961; Sakai & Mikkola, 1971) but *direct* evidence for this suggestion was lacking. Dark-field transmission electron microscopy using a superlattice reflection revealed that in these early stages the alloy is composed of small discrete domains of order within a disordered matrix (Sakai & Mikkola, 1971). The ordered domains are