apparently disordered alloy, provided evidence for a *Gibbsian Type I* reaction for the ordering in Cu Au. The significance of such a transformation in the field of disorder → order reactions has been fully documented elsewhere [3], and the present observations support this viewpoint; the growth of ordered domains in a predominantly disordered matrix is clearly visible. This implies a transient two-phase (ordered + disordered) structure during annealing, a fact already established by TEM in a number of superstructures [4, 5]. Domain shape anisotropy is emphasized after 4.5 min at 350° C (Fig. 4), while in Fig. 5 (6 min at 350° C) we can see the commencement of macrotwinning. The final stages of the isothermal reaction consist of coarsening the macrotwins already formed (Figs. 6 and 7), If the sample's temperature is raised to 450° C, i.e. well above the upper critical temperature, then most of the contrast instantaneously disappears as the temperature passes through 385° C. Although Fig. 8 shows some faint signs of contrast, this is due to irreversible surface relief, referred to above, and is not anything associated with residual order. Such a drastic (discontinuous) change in the sample's morphology as it traverses through T_c is a strong qualitative indicator of a first-order transformation. (A "first-order transformation" is a thermodynamic concept and is not to be confused with a "type I transformation" which refers to a particular kind of mechanism). For comparison, the appearance of the same area as in Fig. 8, before raising the sample's temperature from 350 to 450° C, is shown in Fig. 9.

Hence, the PEEM has proved to be a useful technique in providing qualitative morphological data in following the order/disorder transition in CuAu. The approach has provided us with strong evidence of a nucleation and growth (type I) mechanism as a superlattice forms in CuAu and also indicated that the disordering transformation at T_c is a first-order one. The unique feature of these observations is the fact that changes in morphology of a particular field could be followed as they were taking place. This cannot normally be achieved by either optical microscopy (insufficient resolution in the early stages) or TEM (too small a field of view). However, the PEEM is about 30 times more expensive than a high quality optical microscope, and so the improved performance is dearly bought.

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Correction for the effect of electron beam heating on Kossel pattern lattice spacing results

One of the chief uses of the Kossel diffraction technique has been the precise determination of lattice parameters. Since, however, the technique requires the specimen to be directly bombarded with electrons there is a heating effect which results in a systematic error in the calculated parameter. Heise [1] not taking this into account obtained an erroneous result for the lattice parameter of nickel, and other workers [2] underestimating the heating effect obtained a similar value. These results did not agree with those calculated from X-ray diffractometer measurements [3]. Mackay [4], using the pseudo-Kossel technique in which the X-ray

source is not within the crystal under examination and no heating effect occurs, produced results for the lattice parameter of nickel in agreement with earlier X-ray methods. It is not always possible to use the pseudo-Kossel technique, particularly when lattice spacings are required of small volumes of a second phase embedded in a polycrystalline matrix and the availability of electron-probe X-ray microanalysers and scanning electron microscopes in many laboratories facilitates application of the back-reflection Kossel technique. This letter presents a simple method for the correction of the systematic error caused by the heating effect when true Kossel diffraction is employed.

If it is assumed that there is a point source of heat at the point of impingement of the electron beam on the specimen [5] the temperature rise $\Delta\theta$ at a point a radial distance r from the centre can be calculated from the equation:

$$\Delta\theta = \frac{W}{2\pi JC}\left(\frac{1}{r} - \frac{1}{R}\right) \quad (1)$$

where W is the wattage of the electron beam, C the coefficient of thermal conduction of the specimen, J the Joule conversion coefficient (4.18 J cal^{-1}), and R is the dimension of the specimen. Errors have arisen when investigators have made erroneous estimates of the distance r from the point of incidence of the electron beam within which it is expected that the diffraction of the excited X-rays will occur. Since the size of the specimen used in an electron probe microanalyser is typically of the order of 1 cm^3 and r is anticipated to be in the range of microns the term $1/R$ is very small relative to the term $1/r$ and can be ignored.

The approach put forward in this letter is simply the calculation of the lattice parameter from Kossel patterns taken at different beam wattages. If without heating, the true lattice parameter (neglecting other sources of error) is a_{true} and with beam heating it is a_{calc} then:

$$a_{calc} = a_{true}(1 + \alpha\Delta\theta) \quad (2)$$

where α is the coefficient of linear expansion of the crystal under examination. On the basis of Equation 2 there should be a linear relationship between a_{calc} and W with intercept at a_{true}

$$a_{calc} = a_{true}\left(1 + \frac{\alpha W}{2\pi JCr}\right). \quad (3)$$

Furthermore a value can be calculated for the distance r.

To test this method the spacing of (220) planes in one grain of an annealed, polycrystalline specimen of Swedish iron was calculated from Kossel diffraction patterns exposed with different beam wattages. The method of Fisher and Harris [6, 7] was employed for the analysis of the diffraction patterns. The same set of (220) planes in the same grain was studied in each experiment. Three different electron beam wattages were used and four determinations of the lattice spacing were carried out at each wattage. The result of these experiments is shown in Fig. 1 and the experimental errors at each point on the graph are indicated. The mean calculated lattice spacing increases as the beam wattage increases and in keeping with the theory the points lie close to a straight line. The intercept at 1.0138 Å is the calculated (220) lattice spacing of the Swedish iron after correction for the heating effect and the error in the result is indicated by the amplitude of the errors in the calculated lattice spacings. This indicates a lattice parameter of 2.867 Å which agrees well with published data [3].

The results indicate that the procedure described above achieves a more accurate value of the lattice spacing than would be the case if the

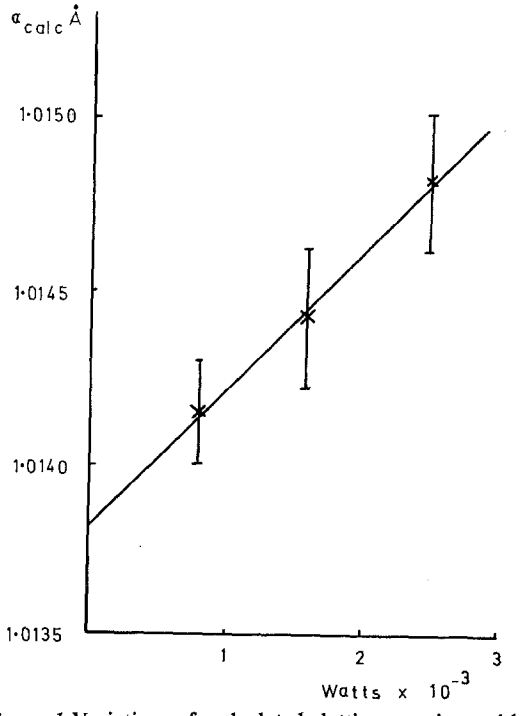

Figure 1 Variation of calculated lattice spacing with electron beam wattage.

heating effect were ignored. An error of 1 part in 10^{-3} was recorded in the calculated value of the lattice spacing due to the heating effect of a 3×10^{-3} W electron beam on Swedish iron. This simple method eliminates a systematic error that always produces a larger value for the lattice parameter than the true value.

Values of $\Delta\theta$ calculated from Equation 2 assuming the thermal expansion coefficient of iron to be 12×10^{-6} give temperature rises of 28, 52 and 87°C at the points on the graph in order of increasing beam wattage. These are the average temperature rises in the volume in which diffraction of the X-rays excited by the electron beam has occurred. Using these values in Equation 1 we calculate a value for r of 7×10^{-6} cm (i.e. 700 Å). From this result it appears that most of the diffraction occurs within a small distance of the incident electron beam. Since there is a temperature gradient in this region there must be a variation in the lattice spacing. This effect must inevitably lead to a broadening of the diffracted X-ray peak and could account for the fact that the familiar white edge (absorption conic) is not seen on (220) Kossel curves from iron grains in back-reflection.

There has been much discussion in recent years about the volume of crystal required for the generation of Kossel patterns. The results of these experiments suggest that it should be possible to obtain Kossel patterns from volumes of less than 1 μm in diameter but there will be some line broadening due to both particle size and temperature gradients. Difficulty arises becauses X-rays not satisfying the Bragg conditions for diffraction at planes in the crystal being bombarded by the electron beam pass on to adjacent crystals and may be diffracted by the lattice planes of these. The resulting pattern is difficult to interpret since it contains a large number of Kossel curves from crystals other than the one directly under examination.

Finally, the temperature rises calculated above are of considerable significance to those wishing to use electron-probe microanalysis for the study of trace elements in plastics or other materials of low melting point. It has often been assumed that coatings are required merely for the purpose of conducting away the incident electrons but in these investigations coatings must also supply good heat conduction.

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Directional coupled growth of a modified Inconel 713C alloy

The morphology of MC carbide obtained by directional solidification of eutectic systems: X–MC where X is Fe, Ni, Co or simple alloys of these metals and M is Ta or Nb was previously investigated [1-4]. Walter and Cline [3] in their work on Co–TaC, Ni–TaC, Fe–TaC, NiCr–TaC, CoNiCr–TaC and FeCrAl–TaC eutectics used hypereutectic compositions in order to produce both primary faceted crystals and fibres of TaC within the same ingot. The study of these faceted TaC crystals enabled them to analyse the factors that determine fibre orientation and morphology, both of which were found to be significantly affected by addition of elements, such as Cr.

The main purpose of the present investigation was to extend these morphological studies to more complex systems and, more specifically, to explore in a preliminary way the dependence of growth morphology of the carbide on growth conditions in a eutectic system consisting of a polynary matrix and a polynary carbide phase.

In a recent investigation [5] of the solidifica-