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THE INTERPRETATION OF ELECTRON DIFFRACTION PATTERNS FROM Ni-A1 MARTENSITE

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Abstract.- Electron diffraction patterns from Ni-A1 alloys have been analysed. It is shown that appearance of extra maxima in the diffraction patterns does not correspond either to a premartensitic precursor phase or a new martensitic phase. Instead they are interpreted as effects arising due to the absolute and relative widths of twins of the 3R-f.c.t. martensitic phase.

Introduction.- Electron microscopy and diffraction techniques enable identification of the crystal structure of the martensite, the lattice correspondence between the parent and martensite phases, and the lattice invariant deformation mode associated with the transformation. The techniques have also been resorted to, often in recent years, in attempting to provide direct proofs of the existence of premartensitic structures and phases. Electron microscopy and diffraction are thus widely accepted now as potent techniques to be used in solving problems related to martensitic transformation and the associated phases and microstructures. Nevertheless, caution must be exercised in proposing the presence of new phases based solely on complexities in the electron diffraction patterns. A careful analysis in such cases taking into consideration, established features related to the transformation may help in arriving at more appropriate conclusions. We elucidate the problem here with particular reference to some conclusions reported by Enami et al. (1) on Ni-A1 alloys.

A Ni-A1 alloys are known to transform to martensite which is twinned and has a Li2-fct-3R structure (2,3). Enami et al. (4) have also shown a softening of the C' elastic constant to precede the martensite transformation and have consequently claimed, based on the analysis of an electron diffraction pattern, the presence of a premartensitic phase with a 7R structure (7). We address ourselves in this report to this latter work of Enami et al.

We intend to show through results from Ni-62 to 66 at % A1 alloys and discussions that the diffraction pattern observed by Enami et al. neither pertains to a premartensitic phase nor would it justify the proposal of a new martensitic phase in Ni-A1.

Results and discussion.- All diffraction patterns presented in this report were obtained from regions that were martensitic. To help in their comparison, most of these diffraction patterns were obtained having the same zone axis with respect to the L12 lattice.

Figure 1a shows the substructure within a martensite plate. It is heavily faulted or twinned. The selected area diffraction obtained from the region is shown in fig. 1b. Enami et al. (1) in showing a similar diffraction pattern have interpreted it as arising from a premartensitic phase with a 7R structure. On the other hand, we have observed that the diffraction pattern corresponds to a completely martensitic region. The appearance of reflections in this pattern that seem to divide the L12-f.c.t.-3R reflections into seven parts does indeed suggest the structure of martensite at first sight to be 7R. In order to confirm whether 7R represents a true phase a diffraction pattern was obtained (fig. 2b) from another area in the same martensite plate but to the left of that shown in fig. 1a, where very few striations may be seen to exist (fig. 2a). This diffraction pattern (fig. 2b) is essen-
Figure 1: Substructure within a martensite plate (fig. 1a) and associated diffraction patterns (fig. 1b).

Figure 2: Different region in the same martensite plate as in fig. 1. Note fewer striations (fig. 2a) and reflections from a L1_0-3R-f.c.t. structure (fig. 2b).
tially that of a simple Li$_2$-f.c.t.-3R structure and exhibits no extra reflections that can be attributed to a 7R structure. Very weak reflections, however, may still be observed in fig. 2b that are located at positions corresponding to an f.c.t. twin. The few striations present in the area of fig. 2a from where the diffraction pattern was taken, would thus appear to be these f.c.t. twins. The following analysis considers in more detail the effect of the twins on the diffraction pattern.

A twinless microstructure will give rise to a diffraction pattern similar to the one shown in fig. 2b. As twins are added to the matrix, the diffraction pattern will change. Such changes were followed by structure factor calculations, along lines suggested by Delaey (5). In the present calculations, matrix layers m(ABC) were considered to be separated by twin layers n(ACB) with and without an intervening layer of stacking fault. Here A, B and C as usual refer to the stacking positions of closed packed planes and m(ABC) and n(ACB), where m and n are assumed to be integers (for purposes of calculation), specify the matrix and twin thickness respectively. The arrangements used for structure factor calculations were thus of the type

\[ m(ABC) \quad n(ACB) \]

and

\[ m(ABC) \quad B \quad n(ACB) \]

Now, when the twins are reasonably thick, say corresponding to a matrix to twin thickness ratio of 2:1 with m = 30 and n = 15, that is approximately true of the microstructure shown in fig. 3a, the observed diffraction pattern consists of sharp twin and matrix reflections (fig. 3b) as is also predicted by the calculations. The importance of the observations and calculations in this case is that no discrete intensity maxima appear between the matrix and twin reflections.

The diffraction patterns of figs. 2b and 3b may be considered to belong to the two extremes of a spectrum of diffraction patterns varying in detail. The intermediate patterns correspond to lower values of n and/or m and differing twin thickness ratios. Some variations are shown in figures 4a and 4b. Fig. 4a represents the case when the matrix to twin thickness ratio is greater than 2:1. With decreasing m in such cases, while the matrix reflections are sharp, the twin reflections are ill defined and even may appear to consist of several less intense maxima. Calculations also indicate that several reflections between those of matrix and twin may appear with a tendency for the intensity maxima to shift away from the twin position towards the matrix reflection. This feature may also be observed in fig. 4a.

The pattern in fig. 4b corresponds to the case when the matrix to twin thickness ratio is 2:1 or smaller. With decreasing m in this case discrete maxima appear in the vicinity of matrix and twin positions and midway between these positions as shown indicated by the arrow in fig. 4b.

When m and n are very small say m = 1 and n = 1, 6 and 7 layer sequences would seem to result following the two arrangements given above. Thus an apparent 7R structure may be produced in local regions where a thin twin is separated from a thin matrix by a fault. A close look at the microstructure in fig. 1a would show that the twin thicknesses are not uniform. In fact this is also reflected by the diffraction pattern (fig. 1b) where twin spots may be seen hidden amongst other reflections. As evidence to our arguments that the 7 layer sequence is only a particular case of arrangement of the matrix and twin, we show in fig. 4c a diffraction pattern following first principles may be regarded as due to a 10 layer sequence. However, if we remember that the martensite is twinned we can explain the diffraction pattern following the present analysis as an example of the second arrangement for m = 2 and n = 1. No separate 10 layer phase need thus be proposed to explain the diffraction pattern.

The present results and analysis may thus be concluded with the following remarks. In plates of twinned martensite, as the twin thickness decreases and its density increases the diffraction patterns will no longer correspond to a simple superposition of the matrix and twin spots. Instead specific diffraction effects can occur that from a structural point of view can be attributed to structures with superperiods. However, it would be misleading to classify them as new martensitic
Figure 3: Thick twins and matrix to twin thickness 2:1 (fig. 3a). Diffraction pattern from the same area (fig. 3b) exhibits sharp matrix and twin reflections.

Figure 4: Other observed diffraction effects from Ni-Al martensites. See text for details.
phases. Further, any claim that the structures represent premartensitic transition structures would also be erroneous in the context of the present results that the twin thickness and density vary from position to position in the same martensite plate. Instead the diffraction patterns in Ni-Al are best attributed to a $L_1_2$-f.c.t.-3R martensitic structure with twin thickness and density varying from one place to another within the same martensitic plate.

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