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PAIRED-MARTENSITE IN Fe-30%Ni SINGLE CRYSTALS

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Abstract.—The variants of martensite forming isothermally in Fe-30%Ni single crystals at temperatures above $M_h$ appear in pairs. This "paired-martensite" was found to be a bulk morphology and one which exhibits strong, non-bursting autocatalysis. Individual pairs are comprised of two lath-shaped wings which meet edge-on along a (100) junction plane, and groups of pairs aligned along the traces of the (100) planes were observed. The major component of the shape-change of a wing was found to be along its long dimension. The role of this shape-change in the autocatalytic formation of the morphology and the relation of paired-martensite to lath and plate martensite are considered.

Martensite in Fe-Ni and Fe-Ni-C alloys generally forms as laths or plates. During the present study of Fe-30%Ni single crystals, however, variants of martensite were found paired in a distinctive chevron-shape and arrayed in long lines across the specimens. As shown in figure 1a, a martensite-pair consists of two wings, each a different variant, meeting at an obtuse angle. When viewed face-on, as in figure 1b, a pair is seen to have a long dimension compared to the wing width. The morphology can be regarded as two lath-shaped wings meeting edge-on along a junction plane, a shape schematically given in figure 1c. The wings are found to be

2-5 μm in thickness, up to 40 μm in width, and 50-200 μm long. The interfaces tend to be ragged, particularly near the tips, and the wing interiors contain no mid-ribs.

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or evidence of twinning. One remarkable feature of this morphology is the alignment of pairs along the trace of a common junction plane. There can be some slight side-to-side deviation of this alignment, but the existence of such lines up to 1.5 cm in length indicates strong autocatalytic couplings between the pairs.

During a study of needle-shaped surface martensite in Fe-Ni single crystals, Klostermann and Burgers (1) and Klostermann (2) also observed paired-martensite. Despite the lack of experimental details in their reports, it is evident from the descriptions and diagrams that the same morphology was found as is being noted here. Klostermann concluded that the junction plane is \{100\}_{fcc}, that the long dimension of a pair is \langle 01\rangle_{fcc}, and that the wings have \{11\}_f{cc} habits. This habit is interesting because the needle-like surface martensite also had a \{11\}_f{cc} habit, and it is not clear whether paired-martensite is simply another type of surface martensite. The purpose of the present work was (a) to determine whether paired-martensite is a bulk morphology, (b) to verify Klostermann's crystallographic results, and (c) to analyze the striking autocatalysis evidenced by this morphology.

Experimental Procedures.—Two single crystals,* grown by the Bridgman technique and furnished through the courtesy of the Pratt and Whitney Aircraft Company and the United Technologies Corporation, were used. To inhibit the formation of surface martensite, the specimens (cut from the single crystals) were electropolished, plated with nickel, and annealed for 1 hour at 1200° C, and then water-quenched. The austenitic samples were then subcooled to induce the martensitic transformation, the latter being monitored by continuous measurement of the electrical resistance. If the undercooling is large, these alloys transform to plate martensite in a burst. However, paired-martensite forms with slow, non-bursting kinetics between -15° C and -25° C (5-8° C above \( M_s \)). For this reason, samples were cooled in 1-3° C steps and held for sufficient time between steps to determine whether the transformation was initiated. After transformation, the specimens were warmed to room temperature and examined metallographically.

The size of the martensite-pairs was found to be too small for precise two-surface analysis of their orientation, and the inaccuracies of pseudo-two-surface analysis by serial sectioning were too large, so an alternative approach was taken. A single-crystal slab of the 30.4%Ni alloy was cut with a \[01\] normal and polished before being cooled to induce the transformation. If the long dimension of the martensite-pairs is indeed \langle 01\rangle, it follows that pairs in four orientations should be found after transformation along the traces of the (100) plane. The surface relief of the martensite was also studied with a dual-beam Micro-Michelson interferometer (5350 Å source) to identify the shape-change of the transformation.

Results.—Surface Transformation: The formation of paired-martensite in samples with heavily nickel-doped surfaces indicates that it is a bulk morphology, like laths and plates, and not a form of surface martensite.

Morphology: As shown in figure 2a, tracks of paired-martensite, some over 1 cm in length, formed along the traces of the (100) planes in the single-crystal slab with the \[01\] normal. The surface relief of the martensite was also studied with a dual-beam Micro-Michelson interferometer (5350 Å source) to identify the shape-change of the transformation.

As previously mentioned, four orientations of martensite-pairs should be observed along the traces of the (100) planes in this sample, if \langle 01\rangle is the direction of the long dimension. One of these pairs should have very thin wings because its junction would be viewed directly end-on, while another should lie flat in the specimen surface. Figure 2b shows that pairs in four such orientations were formed; the types have been identified for the sake of discussion by the inclination of their junction direction to the surface. The thinness of the wings of the perpendicular pairs and the length of the flat ones indicate that the long dimension is \langle 01\rangle or very close to it, a result that agrees with Klostermann (2).

*Fe-29.5%Ni-0.007%C-0.85%Si and Fe-30.4%Ni-0.023%C-0.36%Si-0.007%Cr (wt.%).
Almost-flat

\[ \ldots \]

Almost-perpendicular

Fig. 2: a) Paired-martensite on transformed, prepolished (012) surface; b) Four orientations of martensite-pairs with (100) junction planes.

Figure 3 shows the transformation-produced surface relief about a group of perpendicular pairs and a flat one. The fringe-displacement indicates a depression of the surface in the vicinity of the junction of the outer faces and a rise at the junction of the inner ones, an estimated shear of 15-18%. While precise measurement of the shape-change could not be made because the smallest obtainable spacing of the fringes allowed only two or three fringes to cross the wings of a pair, the interferometric results did show that the major component of the shape-change is along a pair's long dimension. This is confirmed by the deflection of the chance scratch running across the flat pair in figure 3a. Deliberate fiducial scratching of the specimen before transformation was not feasible because such scratches promote the formation of surface martensite.

The lack of a mid-rib or comparable microstructural feature makes the habit plane of a wing difficult to identify; Klostermann did not specify what feature he used. Umemoto and Tamura (3) found two types of paired-martensite in a Fe-Ni-C alloy, one with mid-ribs in the wings and one with no mid-ribs but flat outer faces. They assumed the faces were mid-ribs and used them for analysis. While the outer faces of the paired-martensite considered here are not perfectly flat, they are at least a sharp microstructural feature and so were used in determining the habit.

Since the trace of the normal to a plane will pass through the pole of that plane on a stereographic projection, the angles between the outer faces of 24 perpendicular pairs, 23 almost-perpendicular, and 10 almost-flat pairs were measured. By assuming that the (100) junction plane bisected these angles, the positions of
the normal traces could be specified. The traces for one wing of each type of martensite-pair are plotted in figure 4a. The poles of the faces of the perpendicular pairs lie on the basic circle and have an average value of (342), which is 10° away from the (121) habit of Klostermann.

If the habit plane parallels the outer face, it lies in one stereographic triangle, marked as A in figure 4b for the case of a perpendicular wing. If, however, the habit is actually (121), it could be associated with either triangle A or triangle B, and additional information such as orientation relations or shape-change direction is necessary to make a choice. The orientation relations for the wings of a pair were measured by Klostermann (2) and indicate a habit in triangle B. Martensitic plates whose habits lie in the same triangle have similar shape-change directions near <278> / (4). For example, plates in triangle A have shape-change directions near [278], while those in B, near [278]. The long dimension of a perpendicular pair is [012], and if its wings have shape-changes like those of plates, then the observed shape-change also suggests a habit in triangle B.

Both the measured orientation relations and the shape-changes of the paired wings thus indicate that a distinction must be made between the habit and the outer face. Although the habit and interface plane must lie in different triangles, they are not likely to be too far apart. Since (112) is the closest one can get to (234) and remain in the appropriate triangle, it appears to be a reasonable, albeit tentative, assignment for the habit. The results of the crystallographic analysis are schematically illustrated in figure 5a.

Autocatalysis: Not only does the pairing of martensitic variants indicate a strong interaction between the wings of a pair, but the remarkable alignment of pairs over long distances also points to autocatalysis. Examination of lines of martensite-pairs showed that a single such group is made of pairs in only two of the four possible orientations, the two orientations with the most closely aligned (012) directions. For example, a line of flat pairs in the transformed slab also contained almost-flat pairs but neither of the other two, and vice versa. Figure 5b is a representation of the fretwork that is formed.
The habits of the two types of pairs making up a fretwork are grouped about a common (110) pole. Bokros and Parker (5) observed such groupings in their study of autocatalysis among burst plates and analyzed them as due to the couplings between the elastic stress-field of one plate and the shape-change of another. In this view, potent couplings are those between plates whose shape-changes tend to cancel in a self-accommodating manner. In contrast, the strongest autocatalytic coupling in the paired-martensite case is obviously that between the wings of a single pair, and yet these wings have similar shape changes, not cancelling ones. For a martensitic lath with its transformation strain along its long dimension, the stress in the matrix along the broad faces is opposite in sign to the strain, but directly in front of the lath edges it is of the same sign (7). Thus, if a second martensitic unit were to form in the latter area, one with a similar shape-change would be favored. This is precisely the position and relation of the paired wings.

To examine the potential role of the elastic stress-field about a martensite-pair in creating an alignment of pairs, the field was approximated as that of a set of [012] screw dislocations arrayed around the periphery of a martensite-pair. The Burgers vector was adjusted to produce a -15% transformation strain. The resulting shear stress that would couple most strongly with the shape-change of the next martensite-pair, \(\gamma_{yz}\), is shown in figure 5c. As can be seen, the maximum stress is reached in the vicinity of the junctions of the faces. The largest highly-stressed region is located between the wings; the greatest autocatalytic assist, then, would be given to another pair if it formed in this area, had the opposite shape-change, and lay fairly parallel to the first pair. This suggests that a family of martensite-pairs may propagate preferentially in one direction.

Discussion.- Paired-martensite has been noted previously (7-13), mostly as a minor constituent of burst microstructures where small chevrons are found around the periphery of the plates; such microstructures were also observed in the present study. Little attention has been paid to paired-martensite in these cases, but what analysis has been done indicates that the pairs have (100) junction planes and interfaces parallel to \((225)\) and \((259)\) which are known plate habits. Depending on the alloy system, pairs with internally twinned wings or simply dislocated ones have been identified. No analysis of the three-dimensional shape of these pairs appears to have been conducted yet. Umemoto and Tamura (3) studied paired-martensite in Fe-Ni-C and Fe-Ni-Cr-C alloys under conditions where it was the only type of martensite to form. Such pairs displayed mid-ribs and internal twinning and had the shape of two half-lenses meeting along a (100) junction plane. The habits were near \((225)\).

Several names have been applied to the paired morphology—butterfly martensite is one of the more frequent—but none of these names fully describes the different three-dimensional shapes of paired-martensite in the systems where they have been determined. A feature common to all appearances, however, is the pairing of the variants, and so the name "paired-martensite" is chosen here to describe the morphology. The significance of the widely observed (100) junction plane is still not obvious, but it is clearly an important feature of the pairing.

Inasmuch as paired-martensite is a bulk morphology, its relation to the better-known lath and plate morphologies must be examined. Is it a distinct morphology or simply a pairing of the other two? Clearly it shares features with plates, and the morphology described by Umemoto and Tamura may be visualized as a pairing of plates. However, the pairing in the present study is not of two normal laths, since laths are thought to have habits closer to \((111)\). Nevertheless, there are indications that this type of paired-martensite may be related to lath martensite, such as the fact that it forms in these single crystals in the same temperature range where lath martensite has been observed in Fe-Ni polycrystals (14). Thus far we have treated the wings of a martensite-pair as single entities. However, the wings are often ragged, particularly near the tips where they seem to break up into smaller units. This is demonstrated in figure 6a where the wing of a pair changes from a single unit to a set of smaller units that look more like lath martensite. A similar effect can be seen in figure 6b. Here the martensite-pairs again lie on the (100) planes, but their ragged tips break up into smaller lath-like units. If paired-
martensite nucleates lath martensite at its tips, its formation may hold important clues for understanding lath martensite.

Conclusions.-

1) Paired-martensite is a bulk morphology in Fe-30\%Ni, like laths and plates. It forms isothermally at temperatures 5-8° C above $M_b$.

2) A martensite-pair is made of two lath-shaped wings which meet edge-on along a (100) plane and have their long dimension along $<012>$. The wing habits seem to be near (112), while the outer faces are closer to (234).

3) The shape-change of a martensite-pair was examined for the first time. The wings have similar shape-changes with the major component along the long dimension of the pair.

4) The fretworks of paired-martensite, which form along the traces of the (100) junction planes, are comprised of pairs in two orientations. The habits of the pairs are grouped about a common [110] pole and their shape-changes couple autocatalytically. The fretworks can extend for long distances in single crystals.

5) Paired-martensite has also been observed in intimate relation with plate and lath martensite.

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