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Low Temperature Martensitic Transformations in In/Tl Alloys

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The fcc to fct martensitic transformation in In/Tl solid solutions has been studied by interference contrast microscopy over the range of temperature from room temperature down to 9.5° K. The form of the initial martensite bands, and the subsequent progress of the transformation, both depend on whether the M_s of the alloy lies above or below 0.5 melting point (° K). The transformation is reversible on heating, and the associated thermal hysteresis is found to increase when the amount of Tl in the alloy lowers its M_s temperature below 0.5 of the melting point. These effects are discussed in terms of the effect of temperature on the strains inherent in the transformation.

1. Introduction

The fct phase formed by the martensitic decomposition of the high temperature fcc structure in In/Tl alloys is a so-called "banded martensite". As this name implies, these phases form as parallel-sided bands of opposite surface tilt and each band is itself sub-divided into fine lamellae, which are often too small to be resolved in the optical microscope. The morphology of these martensites is associated with low accommodation strains across the coherent interface between the parent and product phases. The transformations occur during cooling and usually go to completion within a few degrees of the temperature of initiation referred to as M_S. The lack of a distinct two-phase region between the fcc and fct In/Tl phases, at temperatures near 400° K, caused early investigators [1, 2] to consider the transformation as a second-order, thermodynamic, phase change [3]. This opinion was later challenged when two phases of differing composition were reported after prolonged annealing [4] and by reports of small changes in volume [5], and latent heat [6]. The debate on the thermodynamic order of these transformations has continued, nevertheless, and is now being extended to the phase transformation in ferroelectrics [7], and to the low temperature martensitic transformations [7–10] reported recently in the high field superconductors V_3Si [11] and Nb_3Sn [12, 13].

The question of the thermodynamic order of the phase change in In/Tl alloys is highlighted at cryogenic temperatures because of the obvious absence of diffusion-controlled processes. Luo, Hagen and Merriam [14], for example, have derived what they refer to as an "equilibrium phase boundary" between the cubic and tetragonal phases by studying the X-ray diffraction patterns of a series of alloys at room temperature, 195, 77, and 20.4° K. A further point was obtained at 3° K by extrapolating the linear trend of the superconducting transition temperatures of the strain-free cubic-phase alloys. On the basis of these measurements they conclude that the transformation, when present, always goes to completion, but at the same time they emphasise that the phase boundary should not be taken as an implication that no two-phase region exists. This poses a contradiction, because if the two phases are truly in equilibrium at 3° K, as they suggest, and compositional differences are ruled out by the absence of diffusion, then the phase change must be of second order and no two-phase region is to be expected [3].

The present work was aimed at resolving the question of a two-phase region at low temperatures, by making direct observations of the

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*Current address: Department of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass, U S A microstructure of the alloys. This was achieved with the aid of a specially constructed liquid helium microscope stage [15] which enables the tilted martensite bands to be revealed by interference contrast. The results show that the form of the initial martensite bands and the subsequent progress of the transformation alter when the M_S temperature falls below 0.5 melting point (° K). Below this temperature, the transformation does not go to completion because of stresses set up in the as yet untransformed fcc phase. The thermodynamic order of the transformation is therefore discussed by introducing pressure as a variable, in addition to temperature and composition, when considering the equilibrium between the two phases. According to the binary phase diagrams listed by Hansen [16], the In/Tl system is by no means unique in having a martensite phase labelled as an equilibrium low temperature structure. An examination of the relevant literature reveals that all such systems confirm the present observation in In/Tl that two phases of the same composition can coexist indefinitely if the M_S temperature of the transformation lies below 0.5 melting point.

2. Experimental Procedure

Polycrystalline alloys were prepared from 99.999% indium and thallium supplied by Johnson, Matthey and Co Ltd*, the major impurity in each case being approximately 0.0003% Fe. The toxicity of thallium, and the high oxidation rates of both this metal and indium, necessitates special precautions in the preparation of these alloys. A 100 g master alloy containing 36 at. % Tl was therefore melted under $\frac{1}{2}$ atm argon in a sealed pyrex glass capsule, and subsequently homogenised for 48 h at 140° C. Five-gram ingots of alloys in the composition range 15 to 36 at. % Tl (see table I) were prepared in 5 mm diameter glass capsules by diluting the master alloy with pure indium. After homogenisation the alloys were mechanically polished to 600 paper, strain-relieved by annealing at 145° C for 24 h and then electropolished using the solution recommended by Guttman [1]. Immediately after polishing the specimens were washed with alcohol and the dry surface covered with a thin film of cedar oil to prevent oxidation. Preliminary metallographic examination revealed that all the alloys were single-phase at room temperature.

The alloys were examined over the temperature range from 9.5 to 300° K using a liquid-helium-cooled metallurgical microscope stage described elsewhere [15]. The protective oil film on the surface of the specimens solidified on cooling below about 175° K, but remained optically transparent thereby introducing an additional medium between the sample surface and the objective lens, which considerably reduced the resolution compared to that obtained in earlier studies of low temperature martensites in Cu/Al/Mn and in Ni-steels [15]. Although the oil film often cracked on solidification, this had no effect on the morphology of the martensite, as may be seen in figs. 1 and 3 where the martensite bands pass right through the cracks.

3. Results

3.1. Morphological Characteristics

The morphology of the low temperature martensite was found to depend on the thallium concentration in the alloy. In alloys containing less than 27 at. % Tl, the onset of the transformation was marked by the appearance of parallel martensite bands extending across a grain from one boundary to another, as illustrated in fig. 1a. Subsequent athermal transformation in these alloys proceeded by the formation of new bands, which were usually parallel to the original bands. When the martensite plates were examined at high magnification fine twin bands were revealed within them, as shown in fig. 1b. The morphological characteristics of the martensitic transformation in these alloys are similar to those reported previously for alloys containing 18 to 23 at. % Tl, which have M_S temperatures in the range 300 to 400° K [1, 2, 17].

A marked difference in morphology was observed at high thallium concentrations. As illustrated in fig. 2, martensite bands of differing orientations may nucleate in several parts of the same grain in these alloys and the bands do not in general extend completely across the grains. As the temperature is lowered the transformation proceeds athermally both by the lengthening of the initial bands and by the nucleation of new bands. It is evident in fig. 2b that the transformation remains incomplete at 9.5° K, which was the lowest temperature used in this study.

A further interesting morphological characteristic of the transformation is shown by the photomicrographs in fig. 3, which refer to an alloy containing 27.2 at. % Tl with an M_S

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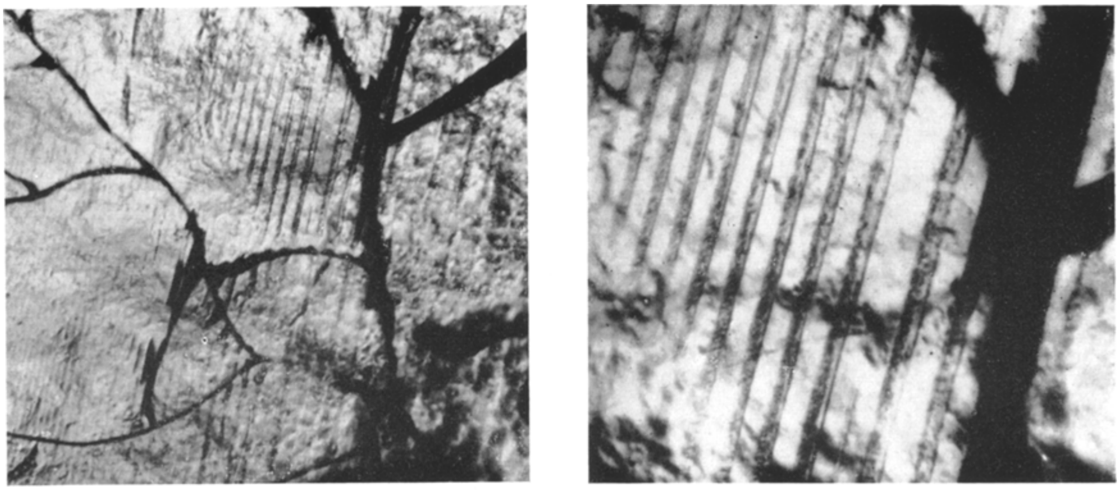

Figure 1 Martensite bands in In/24.4 at. % Tl at 77° K. Interference contrast: (a) ×500; (b) ×900.

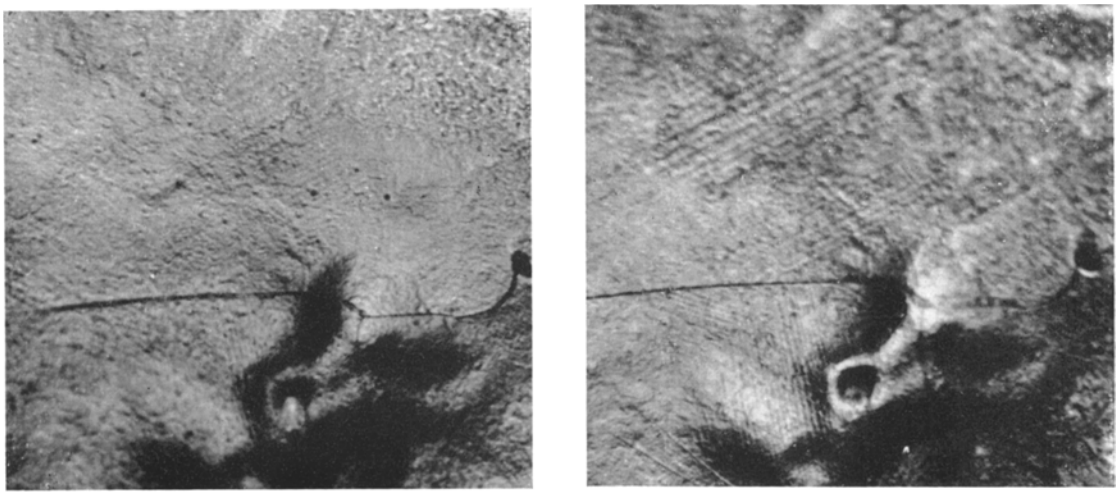

Figure 2 Progress of the martensitic transformation in In/29.5 at. % Tl. Interference contrast: ×500. (a) 25° K; (b) 9.5° K.

temperature of 157° K. When cooled to 150° K, the initial martensite bands, such as those between the two cracks in the oil film, lie approximately parallel to the vertical edge of the photograph. On further cooling to 77° K, however, the orientation of these bands is seen to be rotated through 60°. When the progress of the transformation in this alloy was followed in the microscope during continuous cooling, it was observed that a second set of martensite bands was nucleated at 100° K and grew into the original bands, thereby anihilating them. This effect is reminiscent of the stress induced reversal and orientation changes observed by Burkhart and Read [17] and Basinski and Christian [18] at temperatures above 300° K in single-crystal In/Tl alloys.

In all the alloys studied the martensitic transformation was reversible on heating, the bands disappearing in the reverse order of their formation and orientation changes (if present) also occurring in reverse. It was also found that, provided the temperature on heating was not raised more than about 20° K above A_f, the same martensite bands reappear in the same order on subsequent cooling. Although this cyclic treatment had no effect on the general morphology of the transformations, the M_s, M_f and A_f temperatures of the various alloys were affected as described below.

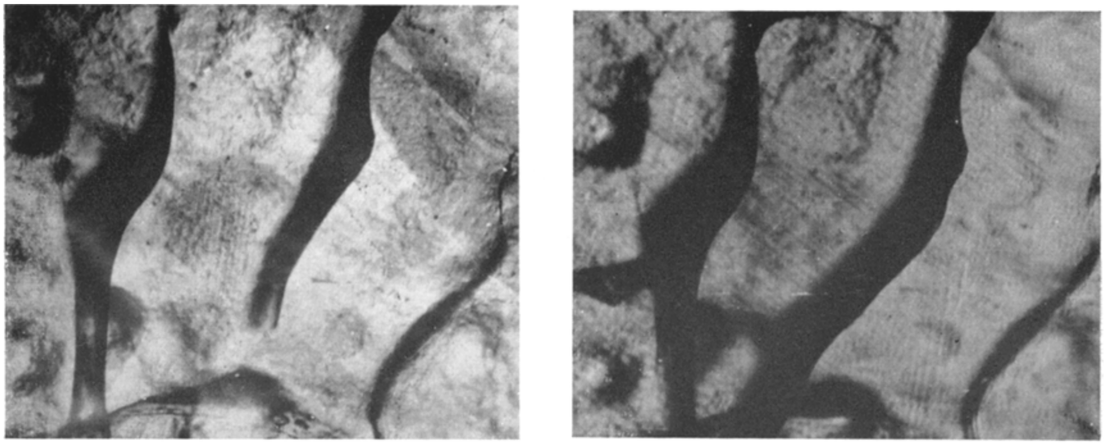
(a) (b)

Figure 3 Change in orientation of martensite bands in In/27.2 at. % Tl. Interference contrast: ×500. (a) 155° K; (b) 77° K

TABLE I Transformation temperatures of In/Tl alloys after successive cooling and heating cycles.

At. % Tl	1st Cycle			2nd Cycle			3rd Cycle		
	M_s(°K)	M_f(°K)	A_f(°K)	M_s(°K)	M_f(°K)	A_f(°K)	M_s(°K)	M_f(°K)	A_f(°K)
18.9	298	—	—						
23.0	298	—	—						
24.2	235	230	240	238	235	245			
25.0	198	190	205	200	185	208			
26.0	178	170	205	185	175	210	186	175	208
26.5	170	165	196	180	173	200			
27.2	157	110	200	170	123	210	171	120	210
28.0	105	60	150						
28.5	110	35	155	120	50	170			
29.0	86	<9.5	146	95	<9.5	150	95	<9.5	153
29.0	90	<9.5	137	98	<9.5	150			
29.2	75	<9.5	110	90	<9.5	120			
29.5	40	<9.5	80	45	<9.5	84			
30.0	20	<9.5	70	40	<9.5	<77			
30.5	15	<9.5	50	25	<9.5	60	25	<9.5	60
31.0	<9.5	—	—						
31.2	<9.5	—	—						
33.1	<9.5	—	—						

3.2. Measurement of M_s, M_f and A_f

The M_S temperatures of the alloys were determined by the first appearance of the martensite when the sample surface was systematically scanned during continuous cooling at a rate of 2 to 4° K per min. Since the time interval required to scan the entire specimen surface with an X 15 (NA = 0.26) objective was less than 1 min, the accuracy of the M_S temperatures for the alloys listed in table I is considered to be ± 2° K. When these M_S temperatures are plotted against thallium concentration as in fig. 4, they continue the decreasing trend established above 300° K by Meyerhoff and Smith [19], and show that beyond about 30.5 at. % Tl, the alloys remain fcc at liquid helium temperatures. The transition temperatures derived by Luo *et al* [14], from X-ray and superconducting transition studies, and the single point reported by Stout and Guttman [5], from a kink in the temperature dependence of resistivity, are also plotted in this figure.

The M_f temperatures of the alloys were taken as the temperature at which the transformation was seen to be complete in the microscope. The results included in fig. 4, show that up to about 26 at. % Tl (M_S = 180° K), the M_S and M_f plots remain approximately parallel and separ-

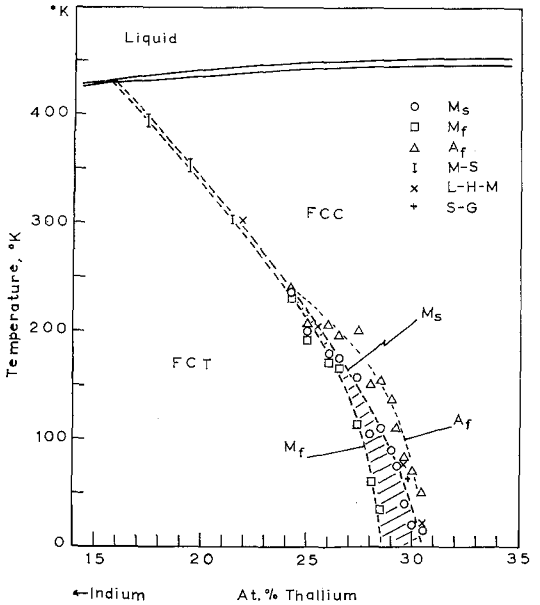

Figure 4 Realisation diagram of the fct to fcc phase transformation in In/Tl alloys. M_s = temperature of transformation on cooling; M_f = temperature of completion of transformation on cooling; A_f = completion of transformation in the reverse direction on heating; M-S [19]; L-H-M [14]; S-G [5].

ated by about 6° K (see table I). At higher thallium concentrations the transformation interval progressively increases and alloys containing more than 28.7 at. % Tl remain incompletely transformed at liquid helium transformations.

The thermal hysteresis of the reverse transformation was studied by measuring A_f, the temperature at the disappearance of the last martensite band on heating. As mentioned above, the transformation is exactly reversible and so the last bands to disappear on heating were those which appeared first on cooling to M_s. The A_f results are also plotted in fig. 4 and show that the thermal hysteresis increases up to a maximum value of 40° K at about 27 at. % Tl. Alloys containing 28.7 to 30.5 at. % Tl, which remain incompletely transformed at 9.5° K, show a slightly lower hysteresis interval.

The M_s, M_f and A_f temperatures of the alloys were all found to be increased when the alloys were cooled and heated a second time, after holding for at least 15 min at a temperature 10° K above their respective A_f temperatures. It is interesting to note, however, that the transformation interval and the thermal hysteresis in a given alloy remained unaltered by the cyclic treatment, as shown by the data in table I. Up to about 26.4 at. % Tl the transformation temperatures were raised by about 5° K while at higher thallium concentrations (i.e. in alloys with a large transformation temperature range) the increase in M_s, M_f and A_f varied from 5 to 20° K. No further changes in M_s, M_f or A_f were observed during a third cooling and heating cycle in alloys containing 26.0, 27.2, 29.0 and 30.5 at. % Tl (table I). The In/27.2 at. % Tl alloy was subjected to two further thermal cycles and still no change was observed in any of the three transformation temperatures, indicating that any plastic deformation accompanying the transformation is not accumulative.

4. Discussion

4.1. Effect of M_s Temperature on Morphology and Growth of Martensite Bands

Since the increase in transformation temperature interval observed in alloys containing more than 26.5 at. % Tl is accompanied by a change in morphological characteristics, a common explanation must lie in the increased resistance to the lengthwise growth of the bands so that they no longer extend right across the grains of the fcc phase. Basinski and Christian [18] have shown that the transformation is very nearly, but not completely, thermoelastic in In/Tl alloys with M_s temperatures near 400° K. Some slight plastic deformation must therefore occur in the as yet untransformed fcc matrix because of the stresses involved in maintaining the coherent interface. At 400° K the ratio of M_s to melting point is greater than 0.8 and hence it is reasonable to expect that the plastic deformation accompanying the transformation will recover continuously, so that the transformation approximates to thermoelastic behaviour. Since the effect of increasing thallium concentration is to lower M_s, the ratio M_s to melting point falls below 0.5 at 27 at. % Tl and hence continuous recovery of the matrix will become increasingly more difficult. The growth of existing martensite plates in these alloys may thus be inhibited by plastic deformation ahead of the interface and the temperature must be lowered, by increasing amounts, to provide an additional driving force before the transformation can proceed by the further growth of the plates or the nucleation of new bands.

The above hypothesis relating the transformation characteristics to the ratio M_s to melting point is supported by the increasing deviation from thermoelasticity observed with decreasing M_s temperature, as indicated by the thermal hysteresis results in fig. 4. The slight reduction of the thermal hysteresis interval observed in alloys containing 29 to 30.5 at. % Tl, may be associated with the incompleteness of the transformation, which causes a reduction in the total deformation.

The formation of the same bands, in the same order, on repeated thermal cycling supports the suggestion of Basinski and Christian [18] that the same nuclei are involved during each cycle and that the fcc matrix phase retains a memory of the transformation to the tetragonal phase. The increase in the M_s, M_f and A_f temperatures can be attributed to an increase in residual stress in the embryonic nuclei or to the creation of new nucleation sites. Both of these effects will be progressively increased by the plastic deformation remaining in the fcc matrix as M_s to melting point falls below 0.5 in alloys containing more than 26.5 at. % Tl. It is not obvious, however, why the hysteresis interval does not progressively increase after repeated thermal cycling as in Fe/Ni alloys [20]. One possibility is that the relatively low melting points of the In/Ti alloys mean that the annealing treatments at temperatures above A_f allow the alloys to recover to an equivalent state of residual stress or defect distribution after each thermal cycle.

4.2. The Thermodynamic Significance of the Two-phase Region

The fact that both the fcc and fct phases are seen to co-exist at temperatures near 9.5° K in alloys containing 28.7 to 30.5 at. % Tl, cannot be used to establish the thermodynamic order of the phase transformation, because the two phases are not in equilibrium in terms of temperature and composition alone. To put it another way, the Lever Rule relating the proportions of the phases to their chemical compositions does not apply in the shaded two-phase transformation interval in fig. 4 because both the fcc and fct phase have the same composition [3].

In contrast to the martensites formed when a diffusion-controlled reaction is suppressed, as in carbon steels or the β-brasses, the fct phase in In/Tl alloys is formed by a shear process even on slow cooling at temperatures in excess of 0.9 of the melting point. It is thus taken to be the equilibrium low temperature structure, as in the phase diagram given by Hansen [16]. Taking all these factors into account, and remembering that no change occurs in chemical composition, the martensitic transformation in In/Tl alloys may be regarded as a polymorphic change in a cone component system, i.e. equivalent to a martensitic change in a pure metal or the melting of a chemical compound.

The metals in which martensitic transformations with well-defined crystallographic relationships have been established are listed in table II. Similar transformations are also considered to occur in Ce, La, Tl and Hf, but no crystallographic data are available for these materials [21]. It is relevant to the present discussion to note that in the metals Fe, Ti, U and Zr, for which the ratio M_s to melting point is greater than 0.5 (see table II), the martensitic transformations occur at a fixed temperature, yet in Co, Li and Na with M_s to melting point less than 0.5 the parent and martensite phases co-exist together over a range of temperature. Further, when the M_s of the fcc to bcc transformation in Fe is lowered to 0.2 of the melting point by the addition of 30 at. % Ni, the transformation can take place over a temperature range of some 130° C, and is accompanied by considerable strain as indicated by a rumpling of the surface [20–22]. Hence the two-phase transformation region clearly represents a metastable equilibrium between two phases of the same composition under conditions of varying temperature and pressure; i.e. the restraining effect of the matrix on the transformation. It is only when such pressure is continuously relieved, by recovery processes, that the transformation appears to occur at a fixed temperature in materials with M_s temperatures above 0.5 of the melting point.

Prolonged annealing (up to 600 days at 350° C [23, 24]) of partially transformed Fe/Ni alloys leads to a breakdown of the coherent interface between martensite and parent phase, together with a change in their chemical compositions so that they assume a new equilibrium in terms of temperature and composition, and thus conform to the Lever Rule. Hence, even though the strains involved in martensitic transformations may be so low that the coherent interface boundaries remain stable indefinitely, published phase diagrams, such as that for In/Tl [16], where phase boundaries are delineated by M_s and M_f plots must not be regarded as *equilibrium diagrams.* This is also of importance in solid solution alloys based on Fe, Ti, U, or Zr, and in other alloys based on In, where the addition of

TABLE II Martensitic transformations in pure metals and some of their primary solid solutions.

Structural change	Pure metals Metal	M_s/ melting point	Solid solutions Solutes	M_s/melting point	References
fcc→hcp	Co	0.40	Fe, Mn, Ni	0.4–0.2	[25–29]
fcc→bcc	Fe	0 75	Cr, Ir, Mn, Ni, Os, Pt, Ru, Si	0.75–0.2	[22, 30–39]
bcc→hcp/fcc	Li	0.12	Mg	0.12–0.24	[40–42]
bcc→hcp	Na	0.09	—	—	[42, 43]
bcc→hcp	Ti	0.55	Al, Cr, Cu, Fe, Mn, Mo, Nb, Ta, V, W, Zr	0.55–0.3	[44–54]
tet→ortho	U	0.66	Cr	0.66–0.44	[55–57]
bcc→hcp	Zr	0.55	Dy, Er, Ho, Nd, Ti, U, Y	0.55–0.3	[52, 58–60]
fcc→fct	In*	—	Cd, Pb, Tl	0.99–0	[1, 2, 4–6, 14, 17–19, 61]

*No martensitic transformation occurs in pure In, but the martensite in In-based alloys has the fct In structure.

the solute lowers the M_s to melting point ratio below 0.5. Some of the relevant solute elements are listed in table II, together with their known effects on M_s to melting point. Following the nomenclature used to describe the continuous cooling or heating diagrams for the Fe/Ni system [16], any such diagrams derived for the above alloy systems should be referred to as *realisation diagrams* and boundaries enclosing metastable regions within which two phases of the same composition co-exist should be clearly labelled M_s, M_f, etc, as in fig. 4.

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