Influence of Order on the Tensile Characteristics of Ni₃Fe

A. E. VIDOZ, D. LAZAREVIĆ AND R. W. CAHN

Department of Physical Metallurgy University of Birmingham, England

Binary alloys of nickel and iron containing 68–80 at.% nickel, and ternary alloys of Ni₃Fe plus up to 6 at.% aluminium, were heat-treated following deformation by rolling or extension, at temperatures just below or just above the critical ordering temperature, $T_{\rm c}$.

All alloys, both below and above T_c , showed a very rapid initial hardening, attributed to short range ordering. The larger the prestrain, the larger was this prompt hardening, which tensile tests showed to be due entirely to a rise in the flow stress.

Alloys near the stoichiometric composition, when prestrained and annealed below T_o , showed a further substantial but slow increase of hardness, greater for larger prestrains. This increase was most pronounced for the 73/27 alloy, which is also the composition possessing the greatest long range order. The ternary alloys, which are known to order more rapidly, also harden more rapidly than the binaries. The slow hardening was therefore associated with long range order; compositions in which no long range order could form underwent recrystallization and softened, but if order developed, recrystallization was inhibited.

Tensile tests showed that the slow hardening, associated with long range order, was not due to an increase of flow stress, but to an enhanced rate of work-hardening. This increase of work-hardening rate is interpreted in terms of a model of the intersections of superdislocations in an ordered lattice, producing tubes of antiphase domain wall which are stretched out between the dislocations after they have crossed. The creation of these tubes requires energy and this causes the increased hardening.

Introduction

This investigation was suggested by a note published by Josso and Waché¹⁾, who demonstrated that nickel-iron alloys at or near the composition Ni₃Fe, rolled and then annealed below the critical ordering temperature, hardened substantially. The hardening was in two stages—an initial rapid rise in hardness was followed by a further slow increase, and was unmistakably connected with the onset These studies have now been of order. extended to a range of compositions, and hardness measurements have been supplemented by tensile tests. In effect, strain-ageing due to crystallographic order has been investigated. A brief account is presented here; a fuller account of the results and of their implications will be published in Acta Metallurgica.

Experimental Methods

Alloys were made by powder metallurgy, and contained 20, 22, 25, 27, 29, 32 and 35 atomic % iron in nickel. Two ternary alloys consisting of 73/27 nickel-iron plus 3 or 6 atomic % aluminium were also prepared. Alloys were rolled into strip; the standard final cold rolling reduction was 80%. Changes in mechanical properties of the polycrystalline alloy on heat treatment were studied (1) by indentation hardness measurements and (2) by auto-recording tensile curves. Some tensile strain-ageing experiments were also performed with alloys of compositions Cu₃Au or CuAu₃.

Results

Hardness Measurements-In Fig. 1, the principal hardness isotherms for a standard annealing temperature of 480°C are assembled for comparison. Individual experimental points are omitted for clarity; the statistical fluctuations in fact never exceeded five points. All the alloys, as rolled, had a hardness close to 300 VPN. All hardened rapidly at first; shorttime tests showed that this rapid hardening was substantially complete within 5 minutes. The long-term hardening was found only in 3 alloys clustered around the mean composition: 73/27. The other alloys softened, and it was established, by X-ray diffraction and micrography, that this softening was accompanied by progressive recrystallization, whereas the

3 alloys which hardened steadily did not recrystallize.



Fig. 1. Hardness isotherms of cold-rolled Ni/Fe alloys annealed below T_{c} .

Existing evidence, from neutron diffraction^{2),3)}, resistivity⁴⁾, dilatometry⁵⁾ and creep⁶⁾, shows with varying degrees of directness that maximum long-range order is produced at or near the 73/27 composition (*i.e.* significantly different from the stoichiometric 75/25 composition), and extends approximately across the range 70–78 at.% Ni. Short range order^{3),6)} is also a maximum an 73-75 at.% Ni, when alloys are annealed above the critical temperature T_c ; this temperature is 503–506°C for the 73/27 alloy, and falls slightly as the nickel content is raised or lowered from this value. Kinetics of ordering were studied by Iida¹⁰, whose calorimetric isotherms above and below T_{e} are in accord with our hardness isotherms obtained under similar conditions. His work shows, as is to be expected, that even below T_{c} short range order forms first, and is superseded only later by the slowly developing long range order.

All these features make it certain that both the rapid and the slow hardening stages are connected with crystallographic order: the rapid stage is due to short range order, the slow stage to long range order. (Isotherms taken above T_o and not reproduced here, show only the rapid hardening). Alloys of compositions which cannot acquire long range order do not show slow hardening.

There is a negative correlation between ordering and capacity to recrystallize. This is attributed to the greater complexity of atomic rearrangement required for an alloy with ordered lattice at the interface between the cold-worked matrix and a growing strain-

free grain; the mean distance through which each atom must move as the interface advances is greater when the new grain has a superlattice than it is when no superlattice is present.

Tensile Tests—Figs. 2–4 show examples of the results obtained in these tests. True stress is plotted against conventional strain, and each curve represents a separate specimen, quenched from 850°C and prestrained by various amounts before the standard ordering treatment.

Fig. 2 shows that the rapid hardening seen in



Fig. 2. Tensile characteristics of 75 Ni/25 Fe alloy, prestrained and given short-term ageing treatment.

Fig. 1 finds its counterpart in a rise of flowstress, which is the greater, the larger the prestrain. The work-hardening characteristics are scarcely affected. The rise in flow-stress is a 'once-only phenomenon'; if a sample is strained and annealed for 2 minutes at 480°C, then strained and annealed again, there is no further rise in flow stress. Again, the rise in flow stress is attributed to the onset of short range order, accelerated by vacancies created by deformations; the larger the prestrain, the more vacancies are available and the more order is created. However, no explanation can at present be offered for the 'once-only' characteristic just referred to.

Fig. 3 shows the long-term ageing characteristics of the 75/25 alloy; again, a long anneal at 480°C was used as standard. For small prestrains, the flow stress still rises after annealing, but the principal characteristic is the pronounced increase in the rate of work-hardening after annealing. After high prestrain, this is the only change, the flow stress remains unaltered. Comparison of Figs. 2 and 3 with Fig. 1 shows the danger of relying exclusively on hardness measurements: there is nothing in Fig. 1 to indicate that, as the annealing time increases, the flow stress at first increases and then drops again while simultaneously the work-hardening rate increases sharply. Either of these increases will result in a rise of hardness, but a tensile test is required to distinguish between them.



Fig. 3. Tensile characteristics of $75 \operatorname{Ni}/25 \operatorname{Fe}$ alloy, prestrained and given long-term ageing treatment.

Fig. 4 shows the long-term ageing behaviour of the 78/22 alloy. This composition is at the margin of the long range ordering field and correspondingly only a slight change in the work-hardening rate is observed. This fact demonstrates that the enhanced work-hardening is due to the onset of long range order. This was further confirmed by exploratory tensile experiments on samples Cu_8Au and $CuAu_3$, which proved to have similar work-hardening characteristics when prestrained and annealed below T_c .

The ternary Ni-Fe-Al alloys (examined by hardness measurement) behaved like the corresponding 73/27 binary alloy, except that the long-term hardening developed much more rapidly in the ternary alloys than in the binary one. This is undoubtedly linked with the fact, deduced from electrical resistivity measurements⁴⁾, that the ternary alloys develop long range order more rapidly. It appears that the aluminium acts purely as an 'ordering catalyst', perhaps by generating additional 'constitutional' vacancies; preliminary X-ray experiments yielded no indication that the aluminium atoms are incorporated into specific sites of the superlattice.

Interpretation

Short Range Order Effect—The rise in flow stress due to the onset of short range order is readily understood in terms of Fisher's⁷ quasi-chemical theory; the replacement of 'right' bonds by 'wrong' bonds, as dislocations cut across a slip plane, requires energy and thus strengthens the lattice.

Long Range Order Effect—Our interpretation of the enhanced rate of work-hardening resulting from the creation of long range order in a deformed lattice is based on ideas developed by Vidoz and Brown⁸⁾. They start from the



Fig. 4. Tensile characteristics of 78 Ni/22 Fe alloy, prestrained and given long-term ageing treatment.

fact[®] that in superlattices, dislocations combine in pairs to form superdislocations. When two superdislocations intersect, the jogs formed in the four participating dislocations will not usually be in line, and the normal consequence will be that as the superdislocations separate after intersecting, they will drag out a tube of antiphase domain boundary between themselves, (Fig. 5). This requires an extra supply



Fig. 5. Production of tubes of antiphase domain boundary by a superdislocation intersecting a forest superdislocation (the latter is not shown).

of energy, and therefore a greater stress is needed to move the dislocations. As the strain increases, more intersections occur per unit of strain, and therefore the extra stress needed increases also. In this way, Vidoz and Brown were able to account quantitatively for the difference in the stress-strain curves of ordered and disordered forms of the same alloy. (It is necessary to make the assumption, which is certainly not the exact truth, that the dislocation patterns, antiphase domains apart, are the same in both forms of the alloy). When a prestrained sample is ordered, existing dislocations become joined in pairs by antiphase domain boundaries. Since such pairs will not normally be coplanar, they cannot glide. New dislocations are created, and—being few—move rapidly and thus make many intersections with the immobile forest dislocations per unit macroscopic strain. In this way the very rapid work-hardening can be understood. No attempt has been made, however, to put this model for order-ageing on a quantitative basis.

References

- E. Josso and X. Waché: Compt. Rend. Acad. Sci. 242 (1956) 1984.
- 2 B. G. Lyashchenko, D. F. Litvin, I. M. Puzei and Yu. G. Abov: Kristallografiya 2 (1957) 64.
- 3 M. F. Collins, R. V. Jones and R. D. Lowde: Proc. Int. Conf. Mag. & Cryst., 1961, Kyoto II (J. Phys. Soc. Japan 17 (1962) Suppl. B-III), 19.
- 4 R. J. Wakelin and E. L. Yates: Proc. Phys. Soc. B66 (1953) 221.
- 5 E. Josso: Compt. Rend. Acad. Sci. 230 (1950) 1467.
- 6 I.I. Kornilov: Mechanical Properties of Intermetallic Compounds (Wiley, New York, 1960)
- 7 J.C. Fisher: Acta Met. 2 (1954) 9.
- 8 A.E. Vidoz and L.M. Brown: Phil. Mag. 7 (1962) in press.
- 9 M.J. Marcinkowsky, N. Brown and R.M. Fisher: Acta Met. 9 (1961) 129.
- 10 S. Iida: J. Phys. Soc. Japan 7 (1952) 373; 9 (1954) 346.

DISCUSSION

Brandon, D. G.: Could some of the peculiar effects seen in the annealing kinetics be explained by changes in the vacancy concentration with (a) alloy content and (b) degree of order ?

Cahn, R. W.: I don't think so. If the vacancy concentration were a function of composition and degree of order and if this were the main explanation of the various kinetics observed, then this could not account for the pronounced differences between the cold-worked and the quenched alloys.

Chikazumi, S.: According to our experience on Ni₈Fe, the effect of ordering on hardening was remarkable for polycrystalline material, but it was not the case for single crystal. This fact can be interpreted in terms of Fisher's mechanism. On the other hand, however, we also observed that the single crystal of Fe₈Al was easily hardened when it was perfectly ordered. In this connection, I would like to ask whether you have tried a similar experiment on single crystal and also I would like to ask your opinion about the reason of such a difference between the two kinds of alloys.

Cahn, R. W.: We did no experiments on single crystals of Ni₈Fe, but we did one tensile strain-ageing test on very coarse-grained Ni₈Fe (\sim 3 mm) (whereas most tests were done on material with a 0.1 mm grain size). The coarse-grained alloy behaved rather similarly to the corresponding fine-grained one. I think that systematic experi-

ments on single crystals of $Ni_{3}Fe$ could be well worth doing. I am most interested to learn of the differences found by Prof. Chikazumi between $Ni_{3}Fe$ and $Fe_{3}Al$, but cannot offer any explanations. I should add, however, that we have not found the mechanical properties of $Fe_{3}Al$ to vary greatly with the degree of order.

PROCEEDINGS OF THE INTERNATIONAL CONFERENCE ON CRYSTAL LATTICE DEFECTS, 1962, SYMPOSIUM JOURNAL OF THE PHYSICAL SOCIETY OF JAPAN VOL. 18, SUPPLEMENT I, 1963

Dynamic Interaction between a Moving Dislocation and Point Defects

J. TAKAMURA

Dept. of Metal Science and Technology, Kyoto University, Japan

AND T. MORIMOTO

Engineering Research Institute, Kyoto University, Japan

It is the purpose of this paper to describe a dynamic interaction of a moving dislocation with point defects, and in particular with impurity atoms, in an otherwise perfect crystal. The dynamic interaction we refer to is such that the kinetic energy of a moving dislocation can be dissipated by the elastic and electrical interactions through the process of exciting the vibration of impurity atoms. The calculation is made by using the method of impulse approximation which was originally developed by Bohr to treat the stopping power of matter for a swift charged particle.

It is shown that such interaction gives rise to a large resistance force for a dislocation moving through the forest of impurity atoms in solid-solution alloys, by which the high flow stress of alloys following the yield point at which the dislocation can become free from the locking due to impurity atoms may be satisfactorily explained.

1. Introduction

With regard to the interactions between a dislocation and impurity atoms, emphasis has been placed on the static cases such as elastic¹, electrical², chemical³ and geometrical⁴ interactions. In these theories except the last one, the yield stress of solid-solution alloy has been primarily interpreted as the stress by which the dislocation can become free from the locking due to impurity atoms. From this it is expected that the free dislocations should move under the stress as low as the Peierls stress immediately after the yield point at which there would be no appreciable increase in the dislocation density. However, in solidsolution alloys such a large yield drop is not actually observed. This makes us imagine that the high flow stress following the yield point in solid-solution alloys is ascribable to somewhat like a dynamic interaction of a moving dislocation with impurity atoms.

Although attempts have been made for the interpretation of the dynamic damping of moving dislocations relating to the internal friction $^{5)-8)}$, these theories are not primarily described in terms of how impurity atoms impede the motion of free dislocations. Recently, Johnston and Gilman⁹⁾ and Johnston¹⁰⁾ have shown that small amounts of impurities controll the flow stress of LiF, and that it is the dynamic resistance to dislocation motion in a crystal that determines the yield stress of LiF crystals. This fact suggests two directions in which we may seek an understanding of the hardening, being due to isolated impurity atoms and to agglomelated defects. In this respect, Fleischer¹¹⁾ has pointed out that a moving dislocation can interact strongly with divalent impurity-vacancy complexes producing large, tetragonal lattice distortions, by which the temperature dependence of the flow stress in LiF was explained. The temperature vari-