

Effects of ordering on the properties of Ni-Cr alloys

ALESSANDRA MARUCCO

*Istituto per la Tecnologia dei Materiali Metallici non Tradizionali,
Consiglio Nazionale delle Ricerche, Milano, Italy*

BIRENDRA NATH

*Central Electricity Research Laboratories, Central Electricity Generating Board, Leatherhead,
Surrey, UK*

A large number of commercial nickel-based alloys are likely to undergo an ordering transformation based on an Ni₂Cr ordered phase formation at temperatures below 550°C. A short-range ordered structure develops in the matrix phase at an early stage of ageing. This can transform to long-range ordering depending upon the composition, after prolonged exposure. The Ni/Cr ratio in the majority of these materials is considerably different from the stoichiometric composition of Ni₂Cr and hence they may not form the long-range ordered phase. Ordering, both short and long-range, is known to have the following consequences: (i) dimensional instability due to lattice parameter contraction; (ii) electrical and thermal characteristics variations; (iii) changes in mechanical properties due to pile ups of dislocation dipoles; (iv) the phenomenon of "negative creep" or material contraction under load.

1. The order-disorder transformation

There exists a number of alloys, falling in between the atomic classification of the intermetallics and the random solid solutions, in which atomic rearrangements in regular patterns occurs below a sharply defined critical temperature, T_c , and the transformation is known as ordering.

Ordered structure arises if atoms of different types are attracted to each other more strongly than atoms of the same type. The ordered phase (or superlattice, or superstructure) forms below T_c at or near simple stoichiometric compositions like AB, A₂B, A₃B, etc. because this interatomic attractive energy exceeds thermal vibration effects. The value of T_c in an off-stoichiometric alloy does not exceed that of the stoichiometric material.

At temperatures just above T_c regular arrays of atomic species do not exist over long-range, i.e. long-range ordering (LRO) does not form but the statistical probability of finding different species as near neighbours may be higher than that in a random solution. This behaviour is known as short-range ordering (SRO).

The majority of the order-disorder transformations in metals occur by a nucleation and growth-type process. The ordered unit cell forms and gives rise to an ordered arrangement of the adjacent atoms. When the ordered arrangement extends over many adjacent unit cells, long-range order is established. The nucleation rate increases with decreasing temperature, but because growth depends on atom diffusion, its rate decreases exponentially with decreasing temperature. Consequently, a classical C-curve kinetics of transformation is observed where the rate of trans-

formation above the nose of the C-curve is limited by the rate of nucleation and that below the nose is controlled by the rate of growth. Nuclei grow at the expense of the solid solution and, in off-stoichiometric alloys, their compositions may be different from that of the matrix. When two domains are large enough to impinge on each other and if their ordered lattices have the same orientation they merge, but if their lattices have different orientation then a translation or rotation antiphase boundary appears. The average size of domains increases with suitable annealing treatments and some regions develop at the expense of others. Depending on temperature and initial concentration, the solution may transform into a two-phase ordered-disordered structure or into a completely ordered alloy. At absolute zero temperature a completely ordered alloy is theoretically possible, i.e. the degrees of LRO and SRO defined by parameters S and σ , respectively, at absolute zero temperature are given by $S = \sigma = 1$ [1-3]. With increasing temperature, S and σ show a behaviour qualitatively represented in Fig. 1. At $T = T_c$, S and σ are generally discontinuous, at $T > T_c$, $S = 0$ and $\sigma \rightarrow 0$. The equilibrium value of S at $T < T_c$, in an off-stoichiometric alloy, is always lower than that of the stoichiometric alloy.

2. Short-range order

SRO can exist in off-stoichiometric alloy compositions where LRO is not possible or at temperatures above T_c for stoichiometric alloys A_xB_y which are long-range ordered at lower temperature. A short-range ordered temperature structure develops in the matrix phase at an early stage of ageing and it can

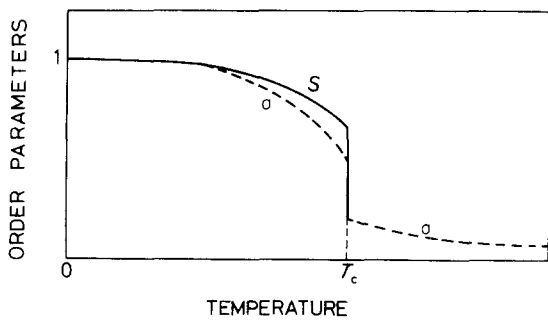


Figure 1 The schematic dependence of the long (S) and short (σ) range order parameters on temperature.

be produced by slow cooling or by annealing after (a) quenching from homogenization temperature, (b) deformation and (c) neutronic irradiation.

Experimental results have led to the formulation of four principal models of SRO [4]:

(a) The statistical model: the SRO parameters α_i represent the probability of finding a B-type atom in the i th coordination sphere around an A-type atom. This probability is independent of the point chosen in the lattice. This SRO model is homogeneous throughout the crystal structure;

(b) The microdomain model: small, long-range ordered domains, which dimensions can reach some tens of nanometres, are separated by antiphase domain boundaries or twin boundaries or other discontinuities. In a stoichiometric alloy, ordered domains can grow until they completely fill the specimen volume and the structure is very similar to a long-range ordered structure below T_c ;

(c) The lattice defect model [5]: dislocations and lattice defects are responsible for the formation and stabilization of inhomogeneities of concentration and degree of order;

(d) The dispersed order model [6–11]: this is characterized by a dispersion of small highly ordered particles, present in a disordered or less ordered matrix, which are resistant to coarsening. The stability of such a non-coarsening structure is possible if the following conditions are satisfied:

(i) there exists a coherent phase boundary on the particles which caused coherency strains due to different atomic volumes of the two phases;

(ii) there exists a particle rim zone in which the local degree of order varies continuously from the value inside the particle to that outside;

(iii) the ratio of specific energy to the product of the specific elastic energy and the rim thickness does not exceed a critical value.

These models differ in homogeneity or heterogeneity and in the SRO stability and the choice between them is sometimes very difficult. The existence of the dispersed ordered structure has been experimentally confirmed in Cu–Al [4], Fe–Al [5], and Ni–Cr [9, 12] alloys.

3. Ordering in the Ni–Cr system

The nature of the equilibrium phase of Ni–Cr alloys has been the subject of considerable study [13, 14] and an order–disorder transformation in nickel-based alloys containing 20 wt % Cr or more is now well established. The matrix phase in these materials forms a short-range ordered structure which can transform to a long-range ordered phase, Ni_2Cr , when exposed to temperatures below 525 to 575°C for long duration, depending upon the composition.

In 1958, Baer [10] showed that a long-range ordered Ni_2Cr phase forms in off-stoichiometric alloys containing 25 to 30 at. % Cr and Bagariatskii and Tiapkin [11] also demonstrated that SRO exists in alloys containing 28 to 35 at. % Cr. SRO has been postulated in a nickel-based alloy containing less than 10 wt % Cr [15].

The kinetics of the transformation are extremely slow and ageing times well over 30 000 h are required in commercial alloys to achieve completion [9]. Studies of ordering in Ni_2Cr have shown that kinetics in the stoichiometric alloy are far more rapid than in off-stoichiometric alloys [12].

In order to obtain a clearer understanding of ordering in Ni–Cr based commercial alloys, studies on the formation of SRO and LRO based on the Ni_2Cr superstructure have been carried out on binary alloys Ni_2Cr and Ni_3Cr . In electron diffraction patterns diffuse scattering and streaking of fundamental reflections in $\langle 110 \rangle$ directions as well as images exhibiting mottled contrast show that a finite degree of SRO forms in both alloys quenched from the disordered state. Both alloys undergo lattice contraction (Fig. 2)

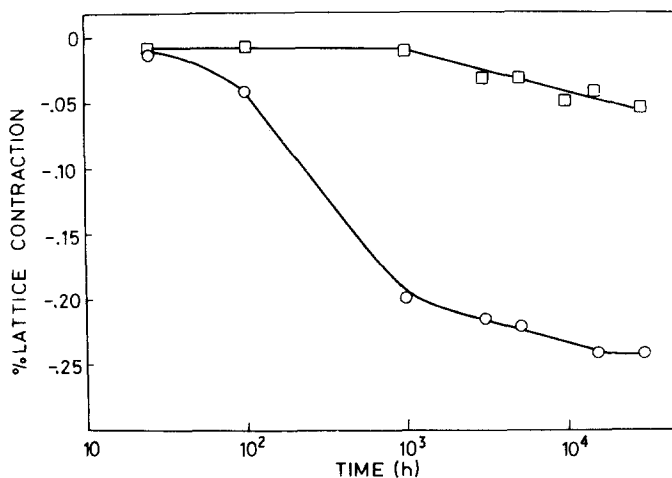


Figure 2 Lattice contraction in (O) Ni_2Cr and (□) Ni_3Cr as a function of the ageing time at 475°C.

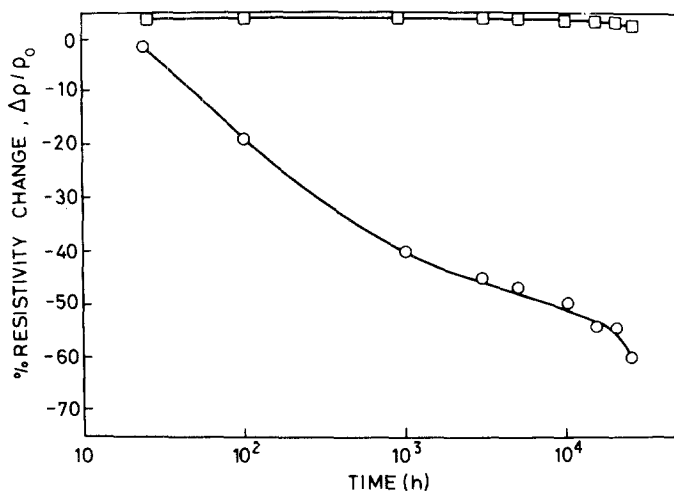


Figure 3 Resistivity changes in (○) Ni₂Cr and (□) Ni₃Cr as a function of the ageing time at 475°C.

and exhibit changes in electrical resistivity upon ageing at and below 550°C (Fig. 3).

The Ni₃Cr alloys showed an increase in resistivity of ~0.4% after 24 h ageing which then remained virtually constant for up to 25 000 h, accompanied by a limited lattice contraction of ~0.05%. The structure remained in the SRO state even after 25 000 h. Neither superlattice reflections appeared nor were any changes observed in diffuse scattering and streaking of fundamental reflections in electron diffraction patterns after 10 000 h ageing, but small coherent precipitates imaged by matrix strain contrast were observed to be uniformly dispersed throughout the sample, providing direct evidence of the dispersed ordered state in these alloys (Fig. 4).

During early stages of ageing the diffraction and the microstructural characteristics of the Ni₂Cr alloy were similar to those seen in the Ni₃Cr material. The SRO state accompanied by an increase in resistivity, begins to transform to a long-range ordered structure after a few hundred hours ageing as proved by Ni₂Cr superlattice reflections in electron diffraction patterns and the concomitant decrease in resistivity. After prolonged ageing a lattice contraction of up to ~0.25% and a decrease in resistivity of up to 60% have been measured. The microstructure of the LRO material reveals both homogeneous (Fig. 5) and heterogeneous

(Fig. 6) nucleation of ordered domains and their subsequent growth.

For example, ageing Ni₂Cr at $T < 525^\circ\text{C}$, where the degree of supersaturation is large, leads to a uniform distribution of ordered domains (Fig. 5). Ordering proceeds by growth of domains until the fields of influence associated with the particles begin to impinge on each other. The results presented here indicate only a slight coarsening of the particulate structure in the long-range ordered Ni₂Cr alloy, with increasing ageing time at a given temperature. Data in the literature [11, 16–21], summarized in Table I, support the findings that the ordered domains attain a size of the order of 20 to 30 nm at $T < 525^\circ\text{C}$ and that growth is slow. At higher temperatures, e.g. 550°C, homogeneous nucleation is difficult because of a low degree of supersaturation. Hence heterogeneous nuclei form in 2000 to 2500 h (Fig. 6) and once formed appear to grow rapidly.

The order–disorder transformation occurs in relatively simple commercial alloys, e.g. Nimonic 80A [9] and also in more complex alloys containing appreciable amounts of other alloying constituents: e.g. LRO phase with the Ni₂Cr structure has been identified in Hastelloy [22]. The critical temperature depends upon the composition.

T_c for Nimonic 80A has been reported to be

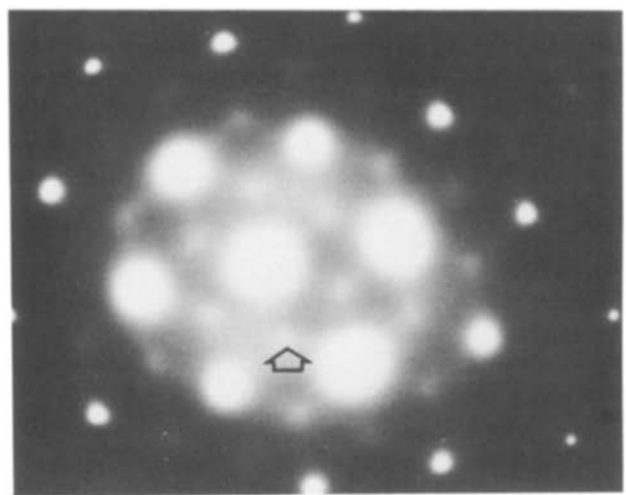
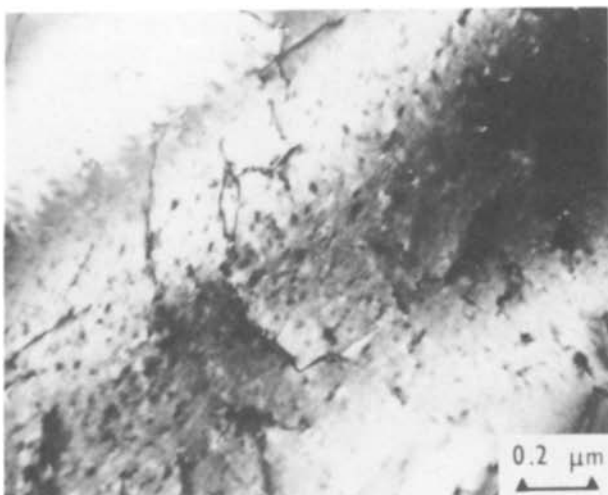


Figure 4 Dispersed ordered state in Ni₃Cr imaged by matrix strain contrast. No superlattice reflection was visible but diffuse scattering (marked by arrow) was present.

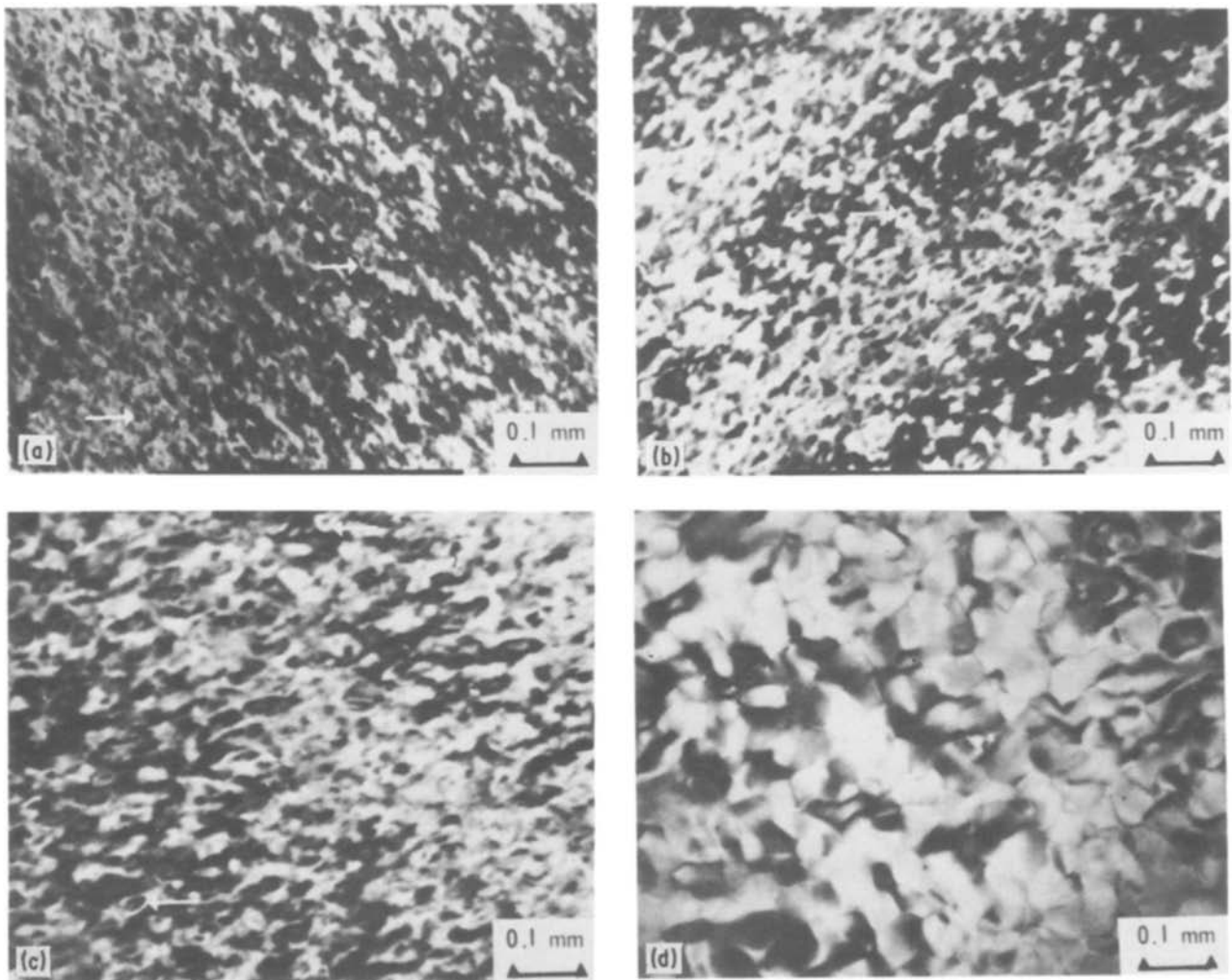


Figure 5 Homogeneous nucleation and growth of LRO domains in Ni_2Cr at $\leq 525^\circ\text{C}$. (a) 475°C , 5000 h WQ; (b) 500°C , 5000 h WQ; (c) 525°C , 3000 h WQ; (d) 525°C , 10000 h WQ.

570°C . This value is in good agreement with the value obtained by ageing a long-range ordered specimen for 1 h at different temperatures (Fig. 7). However, a longer disordering treatment shifts T_c to a lower value (525 to 530°C for 1000 h ageing). A value of 525°C is thus closer to the equilibrium T_c for Nimonic 80A. The discrepancies between the apparent T_c measured after different disordering times reflect the sluggish kinetics of the disordering reaction in off-stoichiometric alloys. The disordering transformation can be regarded as the reverse of the ordering process, whereby Ni_2Cr domains possessing LRO must lose their ordered structure and must disperse excess chromium atoms.

If sufficient time is not allowed for both of these stages to be completed, a metastable degree of order is retained well above the critical temperature.

Property changes at 550°C in Ni_3Cr and Nimonic 80A are due to SRO above T_c . In stoichiometric Ni_2Cr , disordering does not require long-range diffusion of chromium atoms and the T_c of 560 to 575°C is in good agreement with others [12].

4. The K-state

A resistivity behaviour similar to that of Ni_3Cr has been observed in a number of Ni–Cr commercial alloys and it is known as the “K-state”. This state

TABLE I Domain size in Ni_2Cr

Heat treatment	Domain size in Ni_2Cr (nm)	Technique used	Reference
450°C , 100 h	~ 5	X-ray diffraction	Bagariatskii and Tiapkin [11]
500°C , 1600 h	~ 50	TEM	Hirabayashi <i>et al.</i> [16]
500°C , 5 h	$7 \times 3 \times 4$	Neutron diffraction	Vintaykin and Urushadze [17]
10 h	$10 \times 4 \times 6$		
500 h	$12 \times 5 \times 7$		
500°C , 630 h	~ 10	TEM	Klein <i>et al.</i> [18]
500°C , 100 h	15 to 20	Field-ion microscopy	Taunt and Ralph [19]
500°C , 6000 h	(17.5 to 37.5)	TEM	Lepiney <i>et al.</i> [20]
530°C , 856 h	Tens of nanometres (~ 20 to 40)	TEM	Lasserre <i>et al.</i> [21]
540°C , 1100 h	Hundreds of nanometres (~ 170 to 250)		

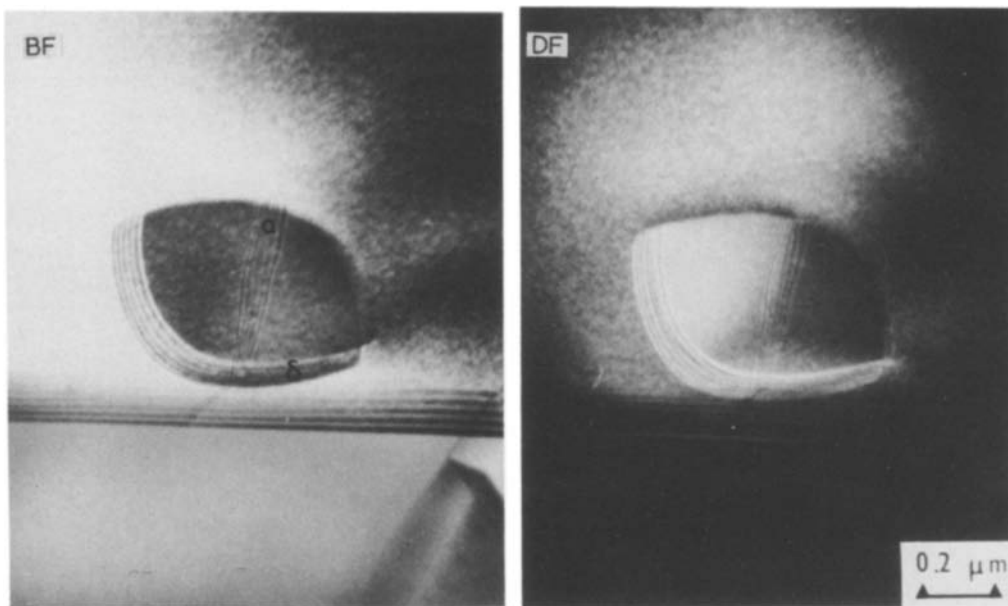


Figure 6 Heterogeneous nucleation of LRO domain in Ni_2Cr at 550°C .

is characterized by an increase in electrical resistivity upon ageing as compared to the quenched state. Further, when a K-state alloy is cold-worked, the resistivity decreases again: this behaviour is contrary to that normally observed in metals and alloys. The anomalous resistivity behaviour, as well as other properties, e.g. heat capacity, thermal expansion, hardness, Seebeck and Hall coefficients, can be attributed to SRO.

The K-state exists in alloys either quenched from high temperatures or cold-worked and then aged at lower temperatures, under the following two conditions [23]:

- (i) the alloy has a transition element as a constituent;
- (ii) the alloy composition is similar to that of the ordered phase.

The resistivity behaviour of some commercial Ni-Cr alloys (Table II) has been studied in order to obtain a better understanding of the ordering reaction, its dependence on composition and its effects on physical properties of Ni-Cr commercial alloys [24]. Fig. 8 shows the resistivities upon 10 000 h ageing.

The study of the anomalous isochronal resistivity

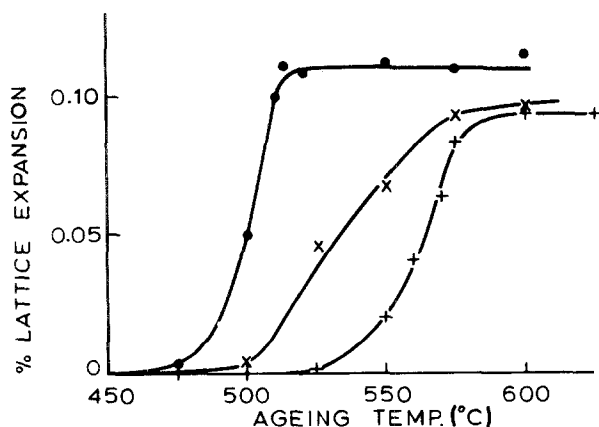


Figure 7 Lattice expansion of Nimonic 80A on ageing pre-ordered (LRO) alloy showing T_c to be 525 to 530°C .

curves (Fig. 9) in TD NiCr led to the following conclusions on the K-state [25].

1. The resistivity of the solution-treated material increases upon ageing due to nucleation and growth of the dispersed ordered state and shows a maximum value when the size of the particles equals the wave length of the conduction electrons at the Fermi level.

2. Furnace-cooled specimen curves do not exhibit this behaviour because SRO is complete in the material before any ageing.

3. The resistivity of the water-quenched specimens is lower than that of the furnace-cooled material because of the disordered structure retained by fast quenching. The resistivity increases as soon as the diffusivity allows short-range ordering.

4. The destruction of SRO during cold working

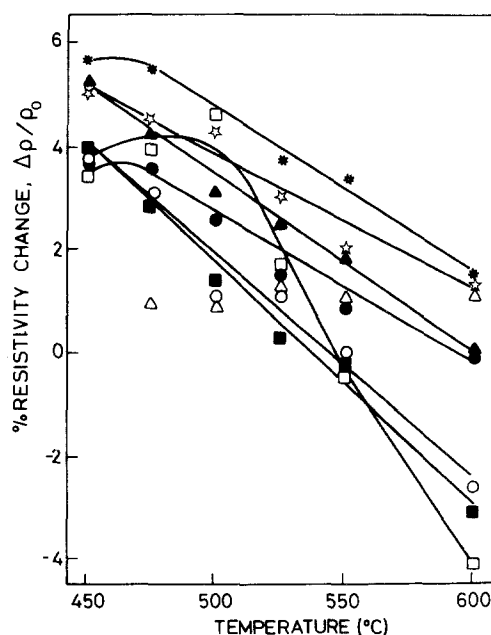


Figure 8 The K-state effects on resistivities of some commercial Ni-Cr alloys upon 10000 h ageing at 475°C . (O) Bright-ray "S", (□) Nimonic 80A, (Δ) 20 Cr/25 Ni steel, (*) Sanicro 71, (●) Inconel 690, (■) Inconel X-750, (▲) Nimonic 75, (☆) TD NiCr.

TABLE II Chemical composition (wt %) of some commercial Ni-Cr alloys

	Cr	Ti	Al	Mn	Si	Co	Mo	Fe	C	Ni	S	Cu	Nb	Nb + Ta	ThO ₂
Brightray "S"	20.0	0.34	0.10	0.04	0.15	-	-	0.02	0.03	Bal.	-	-	-	-	-
T.D. NiCr	19.2	-	-	-	0.003	-	-	-	0.006	78.7	-	-	-	-	2.01
Nimonic 75	19.4	0.36	0.13	0.45	0.53	0.42	0.07	4.28	0.12	Bal.	0.006	0.16	<0.05	-	-
Nimonic 80A	19.45	2.41	1.36	0.07	0.35	0.5	-	0.45	0.07	Bal.	-	-	-	-	-
Inconel X750	14.89	2.50	0.74	0.23	0.23	-	-	6.46	0.03	79.93	0.007	0.03	-	0.93	-
Inconel 690	29.2	0.46	0.26	0.33	0.43	0.03	0.03	8.85	0.01	60.46	-	-	-	-	-
Sanicro 71	15.6	-	-	0.79	0.26	-	-	9.4	0.053	72.7	-	-	-	-	-
20Cr/25Ni-steel	19.8	0.01	0.01	0.77	0.59	0.004	-	53.17	0.022	25.05	-	-	0.6	-	-

is responsible for the resistivity decrease of short-range ordered material. SRO forms in the cold-worked material upon ageing at lower temperatures because the larger amount of point defects which are present in the deformed lattice (interstitials, vacancies, combinations of vacancies, etc.) allow faster diffusion.

5. Negative creep and ordering transformation in Ni-Cr alloys

The expression "negative creep" [26, 27] is used to describe an apparent time-dependent contraction rather than an extension of a sample during creep tests. The phenomenon arises due to a material contraction, caused by structural changes in the alloy and has been observed in a number of Ni-Cr materials. It can be accounted for by considering two opposing contributions to the strain: lattice contraction due to phase transformation, e.g. ordering, and positive creep strain due to the stress. The sum of these two gives the net strain. Thus at high temperatures, just above T_c , and/or high stresses, the positive creep component dominates and negative creep may not be apparent. On the other hand, if the magnitude of the lattice contraction is higher than the creep strain, e.g. at temperatures below T_c , and/or low stresses, then the observed strain is negative. The formation of SRO and LRO based on Ni_2Cr was shown to be responsible for negative creep in Nimonic 80A and Ni-20 wt % Cr at 550°C and below and that the precipitation of the phase γ' , $Ni_3(Al, Ti)$, plays no part in this process [9, 28]. In a constant-strain stress-relaxation test, this

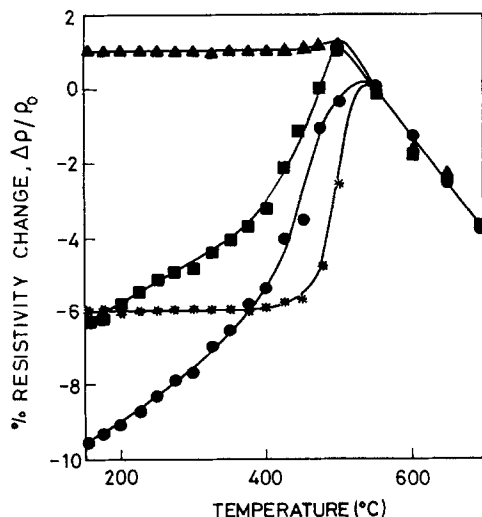


Figure 9 Resistivity curves of TD NiCr upon ageing and cold working illustrate the K-state phenomena. (●) 2.5 h, 1235°C, FC + 50% CW; (■) 2.5 h, 1235°C, WQ + 50% CW; (▲) 2.5 h, 1235°C, FC; (*) 2.5 h, 1235°C, WQ.

is manifested by an increase in stress rather than a decrease [28]. Excessive stresses are generated in constrained components due to lattice contraction associated with the ordering reaction and can lead to failure [29].

6. Effects of ordering on mechanical properties

The existence of an attractive interatomic potential between unlike atoms giving rise to atomic ordering also has a marked effect on the mechanical properties of the alloys, primarily by affecting the dislocation morphology.

In an ordered material where domains are smaller than the width of antiphase boundaries (APB) between pairs of superdislocations, e.g. in dispersed ordered state or during early stages of LRO, the passage of a dislocation along a particular slip plane destroys the unlike nearest-neighbour bonding across the slip plane. Successive dislocations moving on the same plane will be required to produce progressively less disorder: as a consequence the flow stress will drop. Furthermore, because of this increased ease of

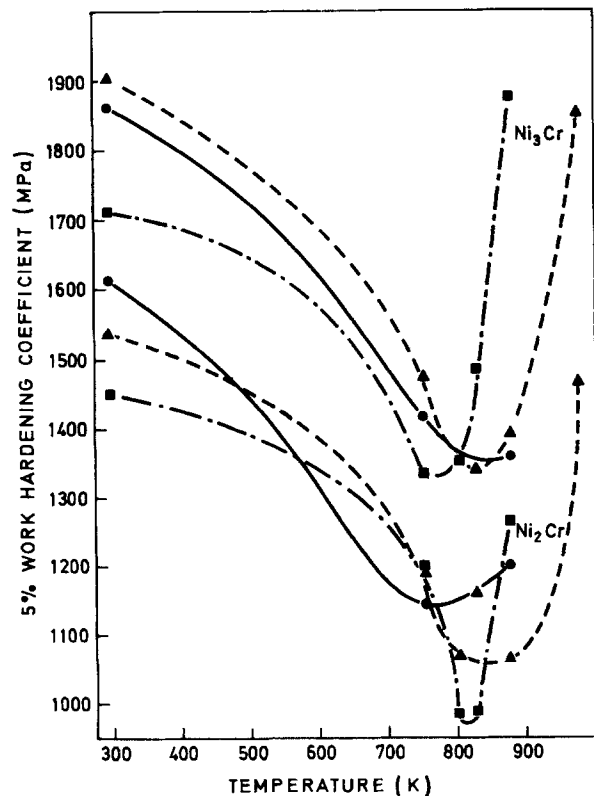


Figure 10 The work hardening coefficient of Ni_2Cr and Ni_3Cr at different temperatures and strain rates. (—●—) $2.33 \times 10^{-4} \text{ sec}^{-1}$, (---▲---) $8.33 \times 10^{-5} \text{ sec}^{-1}$, (- -■ - -) $1.17 \times 10^{-6} \text{ sec}^{-1}$.

dislocation movement on a plane on which slip has already taken place, the deformation is confined to a few slip planes resulting in widely spaced coarse slip steps. Such a behaviour, known as heterogeneous deformation is not observed in pure metals or random solid solutions. If the dislocations were absorbed at the rate at which they arrive at grain boundaries, then the slip will remain planar and no work hardening will occur. If the annihilation was the slower process, then a pile-up will form and further glide on the plane will be restricted and other slip planes will be activated. Glide on different sets of planes will create dislocation tangles and hence, strain hardening.

High-temperature tensile tests have been carried out on Ni₂Cr and Ni₃Cr alloys [30]. The proof stress, the tensile strength and the ductility decrease with increasing test temperatures and/or decreasing strain rate, and the work hardening coefficient shows a minimum between 475 and 600°C, depending upon the test conditions (Fig. 10).

High rates of work hardening are observed at room temperature because of glide of different sets of planes. At higher temperatures higher atomic mobility will have three consequences:

- (i) dislocation annihilation will be faster;
- (ii) the kinetics of ordering in regions undisturbed by glide will increase (but the degree of order below T_c will be of similar magnitude);
- (iii) the process of reordering behind a moving dislocation will be more rapid.

The first two will decrease the work hardening coefficient by encouraging planar slip while the third will have the opposite effect. For a given strain rate, increasing temperature to T_c results in decreasing strain hardening. At temperatures above T_c the expected degree of SRO is smaller but a metastable state of SRO may persist because of the SRO formed during heating of the specimen through T_c . This will result in a low strain hardening coefficient at high strain rates and a high strain hardening coefficient at slow strain rates, as is observed at 550°C and above in Ni₃Cr. At 700°C neither alloy exhibits planar slip and the strain hardening is comparable to that observed at room temperature.

In addition to the three types of serrated flow (A, B

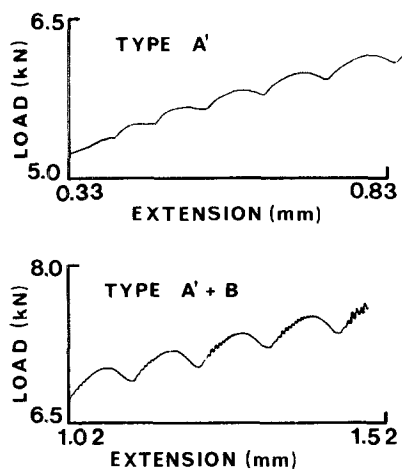


Figure 11 Load-extension traces illustrating a new type (Type A') of serrated flow.

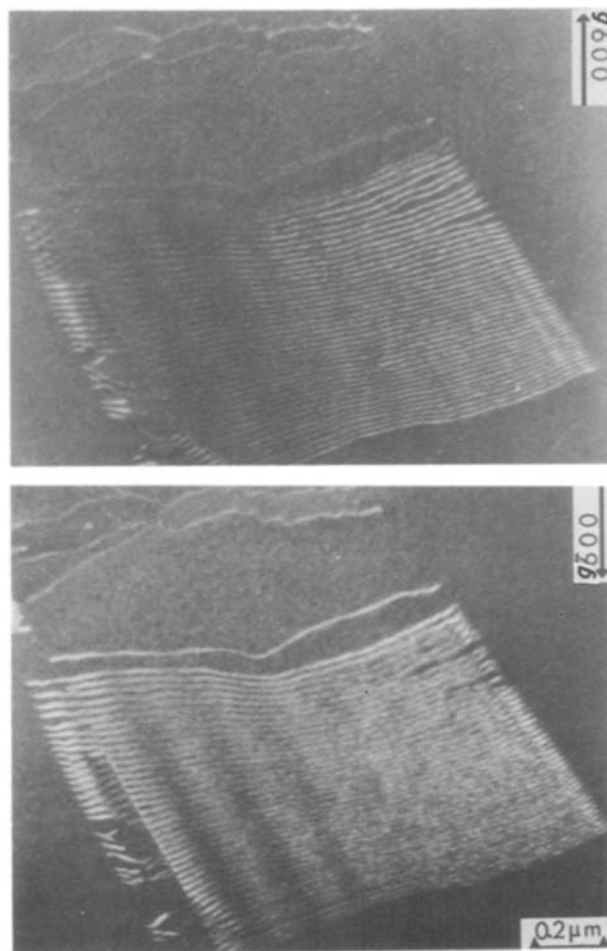


Figure 12 Dislocations multipoles in deformed Ni₂Cr.

and C) associated with dynamic strain ageing [31], a new serration Type A' has been observed (Fig. 11). In Type A' yield, like Type A, there was an initial gradual increase in the flow stress. This was followed by a plateau during the first few serrations of a test and a gradual decrease at higher strains. On continued deformation, Type B serrations appeared superimposed on the Type A' flow. The strengthening during Type A' flow is thought to arise from the formation of arrays dislocation dipoles (Fig. 12). When the applied stress becomes high enough, the two components of dipoles glide over each other and a gradual weakening occurs.

The other parameter which limits tensile strengths of the two alloys is the ductility. At a temperature of 475°C and a strain rate of $2.33 \times 10^{-4} \text{sec}^{-1}$, the fracture surface exhibits a few smooth intergranular facets corresponding to wedge cracks. With decreasing strain rate and/or increasing temperature the fracture becomes totally intergranular and consists of varying proportion of wedge cracks and cavitated facets. The proportion of the cavitated boundaries increases as the temperature is increased and/or the strain rate is decreased. Similar variations in the amounts of W- and R-type cracks have been observed before [32]. The loss of ductility in Ni-Cr alloys is related to cavitation damage at grain boundaries. Cavities could nucleate on coarse grain-boundary ledges which are a consequence of coplanar slip and have been observed in thin foils.

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