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Shock Deformation of K-state in Ni-Cr Alloys

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ABSTRACT

The change of electrical resistivity was used to study the ordering kinetics and the anomalous resistivity behaviour in Ni–22 wt. % Cr and Ni–30 wt. % Cr (Ni–22 Cr and Ni–30 Cr) before and after shock deformation. Ordering was found to occur in two stages : the attainment of an equilibrium domain size, and the increase of long-range order (LRO) within the domains. Specimens of Ni–22 Cr and Ni–30 Cr in the ordered and disorderd states were subjected to explosive shock loading at shock pressures between 90 and 300 kbars. In the pressure range of 200 to 300 kbars, the resistivity of the ordered alloys decreased sharply. The resistivity of the shock-deformed initially quenched alloys decreased continuously between 90 and 300 kbars. The decrease in resistivity with shock deformation for the quenched alloys was related to the destruction of short-range order (SRO).

§ 1. INTRODUCTION

COMMERCIAL nickel alloys containing chromium have been used as electrical resistance and thermocouple materials for a number of years. There has been much work in recent years in understanding the type of ordering that exists in the Ni-Cr system between 23 and 30 % Cr. X-ray structural studies in this system are difficult due to the similarity in scattering power of the nickel and chromium atoms. A number of investigators (Taylor and Hinton 1952, Manene 1959) have attempted to obtain useful evidence by studying nickel-chromium alloys slightly alloyed with an element such as aluminium that facilitates x-ray studies. The interpretation of these results is open to question because of an additional high-temperature transformation that occurs in the ternary alloy, consistent with the precipitation and the re-solution of a second phase. The x-ray investigation by Baer (1959) has clearly established that a 30% Cr alloy should form LRO if annealed sufficiently long.

The structural condition known as K-state has been extensively studied in Ni–Cr alloys containing 10 to 40 at. % where it occurs to a

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pronounced degree. The K-state phenomenon dates from the work of Thomas (1942) on electrical-resistance alloys. An increased value of the electrical resistivity is the most characteristic change in properties that accompanies the formation of K-state in a suitably treated alloy. Roberts and Swalin (1957) reported the absence of long-range order in a neutrondiffraction study of a Ni-Cr alloy heat treated to produce K-state. Bagaryatski and Tyapkin (1958) studied the diffuse x-ray scattering from single crystals of Ni-Cr alloys and concluded that domains about 50 Å in size approximating the composition of the ordered phase Ni₂Cr are characteristic of K-state. Ordering in Ni-Cr was studied by neutron diffraction employing polycrystalline specimens and single crystals (Gomankov, Litvin and Loshmanov 1962). Reflections characteristic of the Ni_oCr superstructure, linked with the original lattice indicated by Bagaryatski and Tyapkin (1958) were found in the single crystal. No superstructure reflections were detected in the polycrystalline specimens, and this was attributed to the low sensitivity of the powder method employed. From a study of conductivity and Hall constant, Köster and Rocholl (1957) concluded that K-state in Ni-Cr alloys involves shortrange order and a change in the average distribution of electrons and holes in the upper energy levels.

Additional investigations of the spical properties of Ni-Cr alloys have concentrated on the anomalous variation in the temperature dependence of electrical resistivity (Taylor and Hinton 1952, Nordheim and Grant 1953, Selissikiy 1962, Yano 1940). Specific heat determination has indicated an anomalous variation which is also sensitive to the initial heat treatment (Stransbury, Brooks and Arlege 1966).

1.1. Deformation of Ni-Cr Alloys

Recent tensile deformation and neutron irradiation experiments on Ni–Cr alloys have indicated that order-induced hardening is present in Ni–Cr alloys. Selissikiy (1962) studied the strengthening of Ni–Cr alloys after low-temperature annealing. Alloy compositions from 18 to 33% chromium were cold worked 97% and then annealed at low temperatures. Strengthening was observed below the recrystallization temperature of 575°c. At concentrations between 23 and 33% chromium an additional strengthening peak due to short-range order was observed. Neutron irradiation of a Ni–21 wt. % Cr alloy increased the ultimate tensile strength, and promoted further transformation to the K-state in a coldworked and quenched alloy (Astrahantzev and Konnov 1966). An investigation of recovery kinetics in a cold-deformed Ni–Cr alloy revealed three recovery stages. The second recovery stage is associated with migration of vacancies while the third is connected with the migration of interstitials (Karpov and Panova 1963).

In summary, to date, no mechanical property data are available on the effect of high strain rates and the formation of K-state in Ni–Cr alloys.

1.2. Ordering Reactions Induced by Shock Waves

Ordering reactions under shock pressures have only recently received attention. The effects of shock waves on the alloy Cu_3Au have been determined by Beardmore, Holtzman and Bever (1964). These investigators subjected specimens of the alloy Cu_3Au in the ordered and disordered states to shock pressures in the range from 160 to 475 kbar. In the pressure range from 290 to 370 kbar. the degree of long-range order in the ordered alloy decreased sharply. Disordered Cu_3Au under explosive loading behaved in a manner characteristic of FCC solid-solution alloys. Furthermore, measurements of x-ray diffraction on Cu_3Au have confirmed the substantial destruction of long-range order at shock pressures greater than 290 kbar (Mikkula 1966). The destruction of LRO occurs over a limited interval of strain and is in contrast with conventional deformation in which disordering occurs at an approximately uniform rate with increasing strain.

Much more work remains to be done on the effect of shock deformation on the ordering kinetics for metals which undergo short-range and longrange order. In addition, there is lack of understanding of shock-induced changes that take place in ordered alloys. For example, the rapid change of resistivity of ordered Cu_3Au alloys at pressures between 290 and 370 kbar, corresponding to transient strains of 0.18 to 0.21, is indicative of the rapid decrease of order and is not understood at present. The purpose of the present investigation is to determine the effects of shock deformation on K-state formation in two alloys of Ni-Cr (22 and 30 wt. % Cr). Shockinduced changes in the ordering kinetics will be explained from the point of view of the effect of vacancies and deformational defects on the ordering process.

§ 2. EXPERIMENTAL DETAILS

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Ingots of Ni–Cr were prepared both by arc melting and by induction melting. The induction-melted ingots appeared microscopically to be much cleaner than the arc-melted ingots which contained a considerable amount of inclusions as well as gross regions of inhomogeneity. In the preparation of the induction-melted ingots, carboynl nickel was melted in an MgO crucible under vacuum and then hydrogen treated. The hydrogen was pumped out, 1 atmosphere of argon admitted, and 99.99% pure chromium was added. The melt was then poured under an argon atmosphere into a copper mold. Ingots 2 in. in diameter by 5 in. long, made by this process contained $22 \cdot 2$ and $30 \cdot 3\%$ by weight chromium. The ingots were then hot rolled into thin foil. The chemical composition of the alloys used in the present investigation is given in table 1. The Ni-22 Cr alloy contained carbon < 0.002 and oxygen < 0.007 wt. %. The Ni-30 Cr alloy contained carbon < 0.001 wt. %.

Alloys	Chromium wt. %	Nickel wt. %
Ni–22 Cr	22.20	87.79
Ni-30 Cr	30.35	69.64

 Table 1.
 Chemical composition of the nickel-chromium alloys

The Ni–22 Cr alloy contained carbon <0.002 and oxygen <0.007 wt. %. The Ni–30 Cr alloy contained carbon <0.001 wt. %.

The specimens used in the quenching and deformation study were in the form of metal strips 3 mm wide, 25 mm long and 1 mm thick. All heat treatments were performed in quartz capsules evacuated to 10^{-5} mm Hg. The specimens were prepared in four initial heat treated conditions:

- 1 The heat treatment of the as-received material of 4 hours at 1250°c, followed by water quenching, was designed to provide alloys in a condition that closely approximated a random solid solution.
- 2 Additional specimens were annealed at 1250°c for 4 hours and then furnace cooled. This heat treatment allowed the alloys to approach equilibrium, which is the ordered state.
- 3 The as-received alloys were furnace cooled from 1250°c to room temperature then water quenched from temperatures between 50 and 1250°c.
- 4 A final set of specimens was water quenched from 1250°c, followed by isothermal annealing at 350°c for 3 hours.

The ordering kinetics for specimens in the 1 and 3 heat treated conditions were determined by resistivity techniques. The isothermal annealing temperature as well as the peak quench temperature were varied for each alloy. Specimens for resistivity analysis had four contact leads spot welded onto each end of the specimen, an outer pair through which the current was introduced and an inner pair across which the potential drop was measured.

Alloys in the 1, 2 and 4 heat treated conditions were shock loaded at 90, 200 and 300 kbar. Shock deformation was accomplished with the flying plate technique (Duvall and Fowles 1963). In this technique, the driver plate thickness controls the magnitude and geometry of the pressure pulse. The pressure obtained from each shot is determined by adjusting the explosive height, and the driver plate thickness. The pulse duration was maintained constant at 2μ sec. The shock loading assembly had been designed so that lateral motion during relief was prevented, and so that a reflected wave could not reach the specimen.

§ 3. Experimental Results

3.1. Results of the Pre-Shock Quenching and Isothermal Annealing Experiments

By isothermal annealing, and by variation of peak quench temperature, the effect of quench temperature on the degree of order and on the ordering rate was determined. In the furnace cooled state (furnace cooled from 1250°c), Ni–22 Cr had a room temperature resistivity of $110\cdot 2\mu\Omega$ -cm, while the Ni–30 Cr alloy had a resistivity of $111\cdot 5\mu\Omega$ -cm. The resistivity in the initial quenched state (quenched from 1250°c) was $102\cdot 3\mu\Omega$ -cm for Ni-22 Cr and $103 \ \mu\Omega$ -cm for Ni–30 Cr. Figure 1 shows the effect of quench temperature on resistivity for the Ni–22 Cr alloy. It is noted that a 7 % drop in resistivity occurs at approximately 500°c. This drop in resistivity was found at a temperature close to the critical temperature of 544°c obtained by Taylor and Hinton (1952).



Effect of quenching temperature on the resistivity of Ni-22 Cr.

The isothermal ordering rate of the two Ni–Cr alloys was studied as a function of quench temperature in order to determine the temperature dependence of the approach towards the equilibrium state. Figure 2 shows that the ordering rate for Ni–22 Cr increases with increasing quench temperature. Figure 3, however, shows that such behaviour does not hold for Ni–30 Cr. The initial slope of each resistivity-time curve was used as a measure of the ordering rate. The ordering rate was found to (1)

Fig. 1

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Effect of quenching temperature on the isothermal annealing at 150° c for Ni-22 Cr.



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Fig. 3

Effect of quenching temperature on the isothermal annealing at $150^\circ \rm c$ for Ni–30 Cr.

increase when the specimens were quenched from above 560° c, and (2) decrease with increasing chromium concentration. It is noted that both Ni-22 Cr and Ni-30 Cr approach a zero ordering rate at 704° c that is lower than for 648° c. The decrease in saturation resistivity between 648° and 704° c is, at present, inexplicable. A possible explanation may be attributed to anti-phase domain boundaries. In quenching from relatively high temperatures (704° c) anti-phase domain boundaries serve as sinks for defect motion during annealing.

Additional experiments were performed on the Ni–22 Cr alloy in order to determine the role of vacancy diffusion in the ordering process, and in order to determine the number of processes activated during annealing. Ni–22 Cr specimens were quenched from 760°c and annealed at 180°c for 200 min so that the ordering rate was essentially zero. The annealing temperature was then changed to higher temperatures of 225°, 250°, 275°, and 300°c for 5, 4, 3, and 2 min, and then lowered to 180°c again. The decrease in resistivity which occurred at the higher annealing temperature indicates that partial disordering (non-equilibrium vacancies) exists within antiphase domains. The first flattening of the curve indicates the formation of antiphase domains. The results of these experiments are summarized in fig. 4.

After holding two specimens at 180° c for 100 min, the temperature was changed from 180° to 225° c and from 180° to 150° c. As shown in fig. 4, the



Fig. 4

Isothermal annealing at 180°c with subsequent breaks at 225°, 250°, 275° and 300°c (Ni–22 Cr).

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resistivity initially decreased in both cases. Holding the two specimens at 225° and 150° c resulted in a higher resistivity at 225° than at 150° c. These results indicate that two parallel processes with different temperature response are contributing to the resistivity change. The first process is the increase in resistivity with temperature and the second is the partial disordering attributed to a non-equilibrium concentration of point defects.



Effect of long time anneals on the final resistivity and hardness of Ni-22 Cr and Ni-30 Cr.

The final resistivity of the initially disordered specimens (quenched specimens) was determined by long time annealing (10⁵ min) and by measuring the resistivity at temperature. Figure 5 shows a maximum around 350°c for Ni-22 Cr and a decrease between 350° and 400°c. Figure 5 also shows a decrease in resistivity for Ni-30 Cr at 400°-450°C. Annealing at 800°c shows an additional decrease in resistivity for Ni-30 Cr. Hence. there is an inherent difference between the resistivity curves of Ni-22 Cr and Ni-30 Cr. This effect for the Ni-30 Cr alloy was not caused by any permanent change such as the loss of chromium reported by Manene (1959) for Nichrome. Chemical analysis for Ni-30 Cr did not show a decrease in chromium after the long-time anneals. It is of interest to note that the hardness for the Ni-30 Cr alloy increased in two steps which corresponded to the increase and subsequent decrease in electrical resistivity. Only the first increase in hardness was recognized for the Ni-22 Cr alloy (fig. 5). The hardness measurements were taken on the same specimens used for the long-time anneal experiments. The specimens were furnace cooled to room temperature before hardness measurements were taken.

Therefore, the heat-treatment results have indicated the following features of Ni–22 Cr and Ni–30 Cr prior to shock deformation :

- (1) There is an anomalous increase in resistivity with temperature.
- (2) There is an equilibrium domain size with LRO within the domains.
- (3) Ordering occurs through the establishment of antiphase domains with an equilibrium concentration of vacancies within the domains.
- (4) The processes attributed to (1) and (2) operate in parallel with different temperature responses.

3.2. Shock Deformation Experiments

Specimens of Ni–22 Cr and Ni–30 Cr were shock loaded after three types of initial heat treatments :

(a) water quench from 1250° C;

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- (b) furnace cool from 1250° C; and
- (c) water quench from 1250 °c followed by isothermal annealing at 350 °c for 3 hours.

Alloy	Heat treatment	Shock pressure (kbar)	$\Delta ho / ho_0$
Ni–22 Cr	Furnace cool (FC) 1250°c	90	-0.0050
	FC, 1250° c	200	-0.0080
	FC, 1250° c	300	-0.0600
Ni-30 Cr	FC, $1250^{\circ}c$	90	-0.0040
	FC, 1250° c	200	-0.0090
	FC, 1250° C	300	-0.0421
Ni–22 Cr	Quenched (Q), 1250°c	90	-0.0001
	Q, 1250°c	200	-0.0003
	$Q, 1250^{\circ}c$	300	-0.0004
Ni–30 Cr	$Q, 1250^{\circ}c$	300	-0.0003
Ni–22 Cr	Q, 1250°C ; A, 350°C	90	-0.0048
	Q, $1250^{\circ}c$; A, $350^{\circ}c$	200	-0.0063
	Q, 1250°c ; A, 350°c	300	-0.0530
Ni–30 Cr	Q, 1250°C ; A, 350°C	90	-0.0070
	Q, 1250°C ; A, 350°C	200	-0.0075
	Q, 1250°c ; A, 350°c	300	-0.0350

Table 2. Shock induced resistivity changes

Table 2 and fig. 6 summarize the shock-induced changes in electrical resistivity of Ni-22 Cr and Ni-30 Cr. It is noted that shock loading at 300 kbar generally destroys the ordered state for (1) the furnace cooled specimens and (2) the quenched and re-annealed specimens. The resistivity of the disordered (quenched) alloys was found to decrease continuously with shock pressure. This change in resistivity is surprising since the

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Effect of shock pressure on the resistivity of Ni-22 Cr and Ni-30 Cr.

shock deformation of a random solid solution is expected to increase the resistivity by producing a high density of point defects. The decrease in resistivity may be an indication that the quench had not completely destroyed K-state.

Figure 7 shows the effect of isothermal annealing on Ni-22 Cr furnace cooled specimens, shock loaded at 300 kbar. In the temperature range of 300° -400°c, within which the K-state is intensively formed (Nordheim and Grant 1953), the resistivity of the furnance cooled-shock loaded specimens attained their pre-shock value. The increase in resistivity during annealing, which is highly irregular for other alloys is further evidence that some type of order occurred. As shown in fig. 7, a similar effect was observed for the Ni-30 Cr alloy. It is apparent that the principal effect of shock deformation on furnance cooled Ni-22 Cr and Ni-30 Cr alloys is not so much in the accumulation of shock induced defects as in the the transition from an ordered to a random solid solution. The subsequent annealing of the furnace cooled, shock loaded specimens forms a state of order which may be similar to K-state.

Isothermal annealing of the quenched-shock loaded specimens resulted in the recovery of K-state and shock induced point defects. This result is expected since the effect of shock deformation of a random solid solution is the accumulation of point defects and dislocations. In the temperature range of 400° - 500° c the resistivity of the quenched-shock loaded specimens approached the resistivity of K-state. Shock deformation does not inhibit the formation of K-state in Ni–Cr alloys.



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Isothermal annealing of Ni–22 Cr and Ni–30 Cr after shock deformation at 300 kbar.

Figure 8 shows that both the Ni-22 Cr and Ni-30 Cr alloys follow a similar shaped isothermal recovery curve. Defect concentration estimates from resistivity experiments require the identification of recovery stages from isochronal annealing experiments (Christou 1972). In the present work three recovery stages were observed for Ni-22 Cr which are identified as Stages III, IV, and V (fig. 9). The specimen resistivity after shock



Isothermal annealing of Ni–22 Cr and Ni–30 Cr after quenching and shock deformation at 300 kbar.

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Isochronal annealing of Ni–22 Cr after quenching and shock deformation at 300 kbar.

deformation is denoted as ρ_s , then $\rho_s - \rho(200^\circ \text{c})$ is Stage III. $\rho(200)-\rho(350)$ is Stage IV, and $\rho(250)-\rho(\text{anneal})$ is Stage V. Resistivity changes during each stage are shown in table 3. Stage V includes both the recovery of dislocations and the formation of K-state. Isochronal recovery data for the Ni-30 Cr alloys was not taken.

P (kb)	$\begin{array}{c c} \text{Total strain} \\ \epsilon t \end{array}$	$\Delta ho III \ (\mu\Omega - cm)$	$\begin{array}{c} \Delta \rho IV \\ (\mu \Omega - cm) \end{array}$	$\Delta \rho V$ ($\mu \Omega$ —cm)
90	0.015	0.52×10^{-2}	0.50×10^{-2}	1.20×10^{-5}
150	0.045	0.45×10^{-2}	0.40×10^{-2}	1.50×10^{-2}
300	0.070	0.42×10^{-2}	0.25×10^{-2}	1.70×10^{-2}
500	0.097	0.30×10^{-2}	0.20×10^{-2}	1.95×10^{-5}

	Table 3.	Resistivity	changes	in	stages	III.	IV.	and	V	for	Ni-22	Cr
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§ 4. DISCUSSION AND CONCLUSIONS

4.1. Discussion of the Quenching Results

The quench-anneal results described in the previous section have clearly identified three types of resistivity changes. Change 1 pertains to the decrease in resistivity during non-equilibrium cooling. Change 2 is the increase in resistivity during annealing after non-equilibrium cooling.

Change 3 is the subsequent decrease in resistivity for the Ni–30 Cr alloy after long-time isothermal anneals.

The observation of a higher resistivity in the ordered state is probably due to decrease in the effective number of electrons associated with the splitting of the Brillouin zone during ordering (Slater 1951) and to the presence of antiphase boundaries. It does not appear likely that trace impurities present would be responsible for the anomaly. In the case where K-state (clustering) is formed prior to quenching, the equilibrium resistivity increases with increasing temperature. The time to establish equilibrium (K-state) increases exponentially with a decrease in temperature, therefore equilibrium is not attained at low temperatures. As a result, the room-temperature resistivity decreases from its equilibrium value, after quenching from above the critical temperature. Experimental curves as shown in fig. 10 can be obtained (Nordheim and Grant 1953).



Resistivity-temperature curves for short-range order and K-state.

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The approach towards the equilibrium resistivity (Change 2) as shown in figs. 2–5 occurs through an ordering reaction. It is noted that the rate of approach towards the equilibrium resistivity was both temperatureand composition-dependent. In discussing the kinetics of order in the Ni–Cr alloys, the data presented indicate that two stages of growth exist. The initial increase in resistivity is due to the nucleation and growth of antiphase domains. This stage results in the establishment of an average domain size within the specimen. The second stage of ordering probably results in the establishment of LRO within the domains. These considerations explain the maximum of fig. 5. The decrease of anneal temperature leads to a decrease of domain size, and therefore to an increase of domain wall scattering and resistance. At low temperatures the domain-wall scattering should be predominant. The contribution from domain wall

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scattering will peak out at higher temperatures due to an increase in domain size. At higher temperatures, the increased equilibrium LRO will predominate and will further increase resistivity. These two effects lead to an inflection point at 400°c and a decrease in resistivity between $400^{\circ}-450^{\circ}$ c.

Change 3 is marked enough to decrease the resistivity of the Ni-30 Cr alloy after annealing at $400^{\circ}-450^{\circ}$ c for 10^{5} min. This change cannot be explained by precipitation of either the chromium rich α phase or impurities. It is noted that change 3 is accompanied by an increase in hardness. The decrease in resistivity of the Ni-30 Cr alloy has also been associated with a large decrease in lattice parameter (Nordheim and Grant 1953). Such a decrease in lattice parameter gives further support to the theory of ordering. It is doubtful whether the long-range order corresponds to Ni₃Cr. The marked increase in hardness suggests that the ordering corresponds to the CuAu type. It is well known that the CuAu ordering involves a marked increase in hardness (Elcock 1956).

4.2. Discussion of the Shock Deformation Results

Shock deformation of the furnace cooled Ni-22 Cr and Ni-30 Cr alloys at 90 kbar resulted in a small decrease in resistivity. This change may be interpreted as a decrease in the amount of antiphase-domain wall. In addition to this, since dislocations move in pairs through an ordered domain there is also a decrease of short-range order at the boundaries. At 300 kbar an additional large decrease in resistivity was observed, suggesting the destruction of long-range order within domains. These results show that a substantial degree of order is retained up to a shock pressure of 200 kbar followed by a rapid destruction at 300 kbar.

The resistivity of the disordered alloys decreases continuously with shock pressure. Apparently quenching does not produce a random solid solution since some degree of short-range order was maintained after quenching. Similar decreases in resistivity after deformation by conventional techniques have been reported for alloys containing short-range order (Elcock 1956).

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The curve of the resistivity of ordered Ni–Cr as a function of the shock pressure has two distinct parts (fig. 6). The values of resistivity suggest that at shock pressures up to 200 kbars the defects produced are primarily of a structural type with the destruction of LRO a secondary effect. At higher shock pressures, the effects involve a progressive disruption of nearneighbour interactions in the ordered lattice. In addition, the rapid change in resistivity at pressures between 200 and 300 kbars may be indicative of a rapid decrease in the degree of order. The destruction of LRO occurred, however, over a limited region of strain, which is in contrast with normal deformation.

The recovery stages observed for the Ni-22 Cr alloy associates the formation of K-state with Stage V. The formation of K-state is activated

and completed between 350°c and the recrystallization temperature. The temperature of 350°c agrees with the previous investigation of Nordheim and Grant (1953). The exact ordering temperature is, however, a function of the relative rate of heating and the rate of reaction for reversal.

§ 5. Conclusions

During annealing after non-equilibrium cooling, the electrical resistivity of nickel-chromium allovs increases towards an equilibrium value which depends on the previous thermal history. Since the effect occurs for alloys with composition corresponding to Ni₃Cr, there is good reason to believe that a structural change, known as K-state formation, has taken place. Two features of K-state in Ni-Cr are emphasized :

- (1) The existence of a small equilibrium domain size, with LRO within the domains.
- (2) The anomalous higher resistivity of the ordered state than the disordered state.

The present investigation has showed that K-state can be formed in shocked Ni-Cr by isothermal annealing above 350°c.

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