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journal or	Science reports of the Research Institutes,
publication title	Tohoku University. Ser. A, Physics, chemistry
	and metallurgy
volume	6
page range	462-468
year	1954
URL	http://hdl.handle.net/10097/26659

On a New Anomaly in the Alloys of Nickel and Cobalt. II The Cause for the Effect of Magnetic Anneal*

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(Received June 10, 1954)

Synopsis

The effect of magnetic anneal on the magnetic properties of Ni-Co alloys has been determined with 6 kinds of specimens in the form of wire, and the cause of the effect has been considered. It has been found that the effect of the magnetic anneal is more remarkable in the wire-specimens than in the ring-specimens, which was reported in the previous paper, and that the effect is available in the temperature range between the magnetic transformation point and about 400°; for an example, it is most remarkable at about 650° in the alloy of 65% Co. Further, from the measurement of magnetization at high temperatures, it has been concluded that the effect of the magnetic anneal is in close connection with the plastic flow due to the magnetostriction at high temperatures.

I. Introduction

One of the present investigators⁽¹⁾ has discovered previously in collaboration with other workers that a remarkable effect of magnetic anneal could be observable on the ring-form specimens of nickel and cobalt alloys, of which hardly any effect had been observed up to that time. That is to say, the magnetization curve after a treatment in comparatively weak magnetic field will be of two-steps type and the magnetic hysteresis curve will show also an extremely peculiar form. As the magnetic field grows larger, its magnetization curve become steep in slope and magnetic hysteresis curve becomes rectangular in type.

It is very important to ascertain the cause for the effect of magnetic anneal mentioned above, because the existence of superlattice has not yet been observed in nickel and cobalt system. The present investigators, by using wire-form specimens which are easy of heat treatment and measurement, examined the effect in various ways, the results of which will be described in the following sections.

II. Specimens and experimental method

Materials used for preparing specimens and melting method are the same as those in the Part $I^{(1)}$. Melts were solidified in a crucible and forged into cylindrical bars 5 mm in diameter. Then they were made into wires about 2 mm in diameter by means of a swaging hammer. Wires 50 cm in length were cut off

^{*} The 770th report from the Research Institute for Iron, Steel and Other Metals. Read at the 5th and 6th sectional meeting on magnetism of the Physical Society of Japan, Oct. 15, 1948 and Nov. 7, 1949 and at the autumn meeting of the Japan Institute of Metals, Nov. 4, 1949. Published in the J. Japan Inst. Metals (Nippon Kinzoku Gakkai-Si), 17 (1953), 612.

⁽¹⁾ H. Masumoto, S. Inoue, I. Ukaji, Nippon Kinzoku Gakkai-Si, 17 (1953), 607; Sci. Rep., RITU, A 6 (1954), 375.

from them to be used in measurement. Alloys were 6 kinds of pure nickel, 10, 20, 33.43, 40 and 65 per cent cobalt, in composition.

For measurement in annealed state, specimens were prepared from the wires mentioned above by heating them in a non-inductive electric furnace at 1000° for 3 hours in vacuum, and cooling them to room temperature at the rate of 100° per hour. In the non-inductive furnace, the differences of temperature and residual magnetic field were kept less than 1° and 0.01 Oe, respectively, over the whole length of the specimen.

For measurement of the state of magnetic anneal, specimens were prepared each time by heating them at 1000° for 2 hours, then cooling them at the rate of 100° per hour passing through the coils wound on the outer part of the furnace with direct current of proper intensity. The strength of external field applied during cooling was 0.1, 0.2, 0.3, 0.5, 1, 2, 5, 10, 20 and 40 Oe, respectively. Besides, various treatments were given to them in order to determine the temperature range in which the magnetic anneal is most effective, details of which will be described in the sections 2 and 3, Chap. III.

For measurement of magnetism at room temperature, the ballistic galvanometer method was used with the magnetizing coil 1 metre long.

Magnetization at high temperatures was also measured by a ballistic galvanometer method with specimens placed in a narrow silica tube wound with secondary coil, using the furnace described above. Heating and cooling of the specimens were done at the rate of 1 to 2° per minute, and the external magnetic field was 2.6 Oe.

III. Experimental results and their consideration

1. Effect of magnetic anneal on wire-form specimen.

In the case of the present investigation wire-form specimens were used as they were easy to handle in various treatment. First, similar measurements as those with ring-form specimens⁽¹⁾ were repeated with wire-form specimens. Figs. 1 and 2 show the results of measurements of the initial and the maximum permeabilities after the magnetic anneal.

As seen in the figures, the initial and the maximum permeabilities increase, similar to the case of ring-form specimens, after the treatment in magnetic field, the effect of treatment being especially remarkable in the maximum

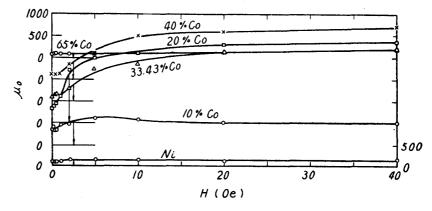


Fig. 1. Relation between the Initial Permeability of Ni-Co Alloys and the Strength of the Field Applied During Cooling.

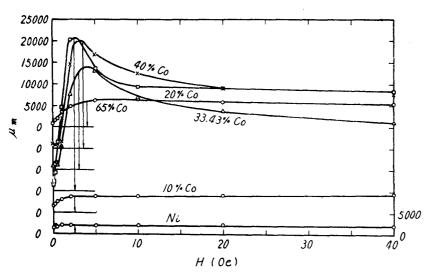


Fig. 2. Relation between the Maximum Permeability of Ni-Co Alloys and the Strength of the Field Applied During Cooling.

permeability. However, the effect is different from that in the case of ringform specimens in the concentration of cobalt range showing remarkable effect, and in nickel, in which the permeability tends to decrease as the applied field grows larger in ring-form specimen, it in-

creases in wireform specimens; it is impossible at present to give any explanation on the cause. Concerning the point that the effect was especially remarkable in wire-form specimens, we should take into consideration the fact that although the demagnetizing factor of the specimens subjected to measurements was as small as 0.0008, the effect of the factor was not yet negligible.

The highest value in the initial permeability was 1850 obtained by cooling the alloy containing 20 per cent of cobalt in the magnetic field of 40 Oe, and that in

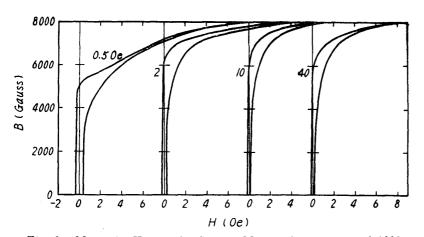


Fig. 3. Magnetic Hysteresis Curves Measured at $B_{\rm max}$ of 8000 Gausses with 20% Co Alloy Cooled in the Field of the Strength Indicated in the Figure.

the maximum permeability was 35,080 obtained by treating the same alloy in the magnetic field of 2 Oe.

In the wire-form specimens containing more than 20 percent of cobalt, the magnetization curves and the magnetic hysteresis curves are of per-

minvar type ⁽²⁾, similar to those of ring-form specimens, when they are cooled in the magnetic field of less than 0.5 Oe (Fig. 3). As the applied field grows larger, the magnetization curve becomes steep in slope and the magnetic hysteresis curve becomes the rectangular type. And where the maximum permeability decreases with increase in the applied field, simultaneously the residual magnetic induction will be smaller, while the coercive force remains almost unchanged.

⁽²⁾ See Part I(1) and J. S. Marsh, The Alloys of Iron and Nickel, Mc Graw-Hill, N.Y., (1938), 269.

2. Relationship between the effect of magnetic anneal and temperature.

In order to determine the temperature at which the effect of magnetic field cooling begins, the specimens was first heated at 1000° for 2 hours and cooled in the magnetic field of 5 Oe at the rate of 100° per hour to a certain temperature, at which they were kept for 1 hour in the field, and after taking off the field

they were cooled again at the same rate to room temperature. In Figs. 4 and 5 are shown the relationships between the increasing rate of the initial permeability, $\mu_{O,T}$, or the maximum permeability, $\mu_{m,T}$, of the alloys treated as mentioned above against those, $\mu_{O,O}$ or $\mu_{m,O}$, in annealed state and the temperatures, T, at which the magnetic field was taken off. And the point A_2 is the magnetic transformation temperature obtained by magnetic analysis (Fig. 6).

As seen from the figure, the effect of magnetic anneal on the initial and the maximum permeabilities generally begins at the magnetic transformation temperature of the alloys and ends at about 400°. And the effect is less in the initial permeability than in the maximum permeability. This fact indicates that the magnetic domain having the orientation in the direction of the axis of the specimen increases by the treatment resulting much increase of maximum permeability, but the magnetic domain under such distribution contributes relatively less to the initial permeability.

The magnetization curve shows the form of perminvar type for 20 percent cobalt alloy when the applied field was

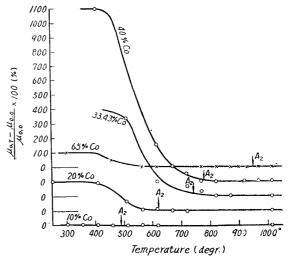


Fig. 4. Relation between the Initial Permeability of Ni-Co Alloys and the Temperatures, at which the Applied Field of 5 Oe was taken off.

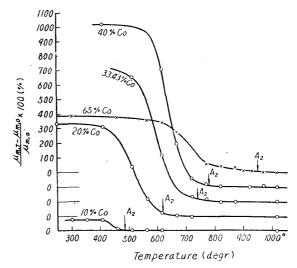
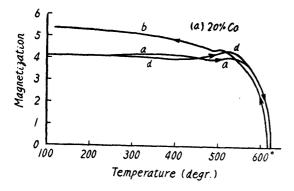


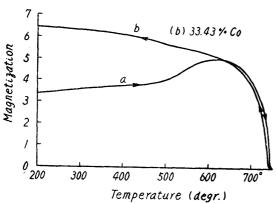
Fig. 5. Relation between the Maximum Permeability of Ni-Co Alloys and the Temperatures, at which the Applied Field of 5 Oe was taken off.

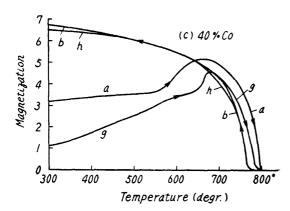
taken off at 550° , for 33.43 percent cobalt alloy at 600° to 700° , and for 40 percent and 65 percent cobalt alloy at 700° to 750° .

3. Results of magnetic analysis

Results of magnetic analysis in the external field of 2.6 Oe are given in Figs. 6(a) to (d). Curve "a" represents heating curve obtained with annealed specimens; magnetization gradually increases, and at about 500° it rises rapidly, reaching a maximum at about 540° in 20 percent cobalt alloy, at about 620° in 33.43 percent







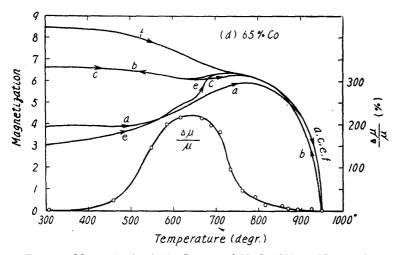


Fig. 6. Magnetic Analysis Curves of Ni-Co Alloys Measured in the External Field of 2.6 Oe.

cobalt alloy, at about 670° in 40 percent cobalt alloy and at about 770° in 65 percent cobalt alloy, and then it suddenly becomes small, finally disappearing at the magnetic transformation temperature. Next, curve "b" represents cooling curve obtained after heating subsequently the specimens up to 1000°. At the magnetic transformation temperature, the magnetization appears, reachs a maximum at a temperature somewhat lower than that in the case of heating curve, and gradually increases to room temperature. The positions of these maxima corresponds to those of steepest slopes in the increasing rate curve of the maximum permeability shown in Fig. 5. Curve "c" represents the results obtained from the measurement in heating made immediately after the measurement of curve "b", and is almost in agreement with curve "b".

Curve "d" in Fig. (a) represents the result of the magnetic analysis measurement in the external field of 2.6 Oe obtained with 20 percent cobalt alloy, after the magnetic field of 13 Oe was first applied on it at each measured

temperature, and then the field reduced to 2.6 Oe. In comparison with curve "a", an apparent rise in the maximum portion can be seen in this curve. Curve "e" in Fig. (d) represents the result of the magnetic analysis in the D. C. field of 2.6 Oe with 65 per cent cobalt alloy after demagnetizing it with

the A.C. field of maximum strength of 13 Oe at each temperature; the maximum becomes very great and extensive in comparison with curve "a". It can be seen thus that when the alloy is once applied with a large magnetic field, whether the field be D.C. or A.C. the effect will appear in response, and that the larger the magnetic field is, the greater the effect will be.

Curve "f" in Fig. (d) represents the results of the magnetic analysis in the external field of 2.6 Oe made in heating with 65 percent cobalt alloy cooled down to room temperature from 1000° in the magnetic field of 40 Oe. The effect of the magnetic anneal becomes smaller from about 500° and corresponds to curve "b" above about 750°. It can be seen, therefore, that the residual effect will be reduced at relatively low temperatures and that a new effect will be added at high temperatures.

Curve "g" in Fig. (e) represents magnetic analysis curve obtained with the wire-form specimens prepared by giving a cold working of 58 percent on 40 percent cobalt alloy. It can be seen that magnetization increases with the rise in temperature, resulting the decrease in residual strain, and it increases still further above 650° , where the effect of magnetic anneal becomes more remarkable. However, in comparison with curve "a", magnetization is smaller because of the residual strain. The curve "h" is the cooling curve after the alloy is subsequently heated up to 1000° , and shows that it almost coinsides with curve "h" because of relieving of strain.

Finally, curve $\Delta\mu/\mu$ in Fig. (d) shows the increasing rate in the maximum permeability against that in the annealed state which was measured with the specimen treated in the following way: The specimen was heated at 1000° , cooled at the rate of 100° per hour to the temperatures in the abscissa, at which the specimen was kept for one hour in the field of 5 Oe, and then cooled again at the same rate to room temperature without the field. It is seen that the magnetic anneal is effective between 400° and the magnetic transformation point, and that the effect is most remarkable at about 650° at which curves "a" and "e" begin to rise.

Two explanations have hitherto been given on the cause for such effect of magnetic anneal on permalloy in iron and nickel system: one finds the cause in the superlattice transformation⁽³⁾, while the other says it is rather due to plastic flow due to the magnetostriction at high temperatures⁽⁴⁾. However, in nickel and cobalt system, any presence of such superlattice has not yet been observed, so we cannot explain the cause of the effect mentioned above with the superlattice transformation. Thus, in the case of nickel and cobalt alloys, it is conceivable that the effect may be caused by the plastic flow due to the magnetostriction at high temperatures, as Borzorth⁽⁴⁾ previously proposed.

Recently an apparatus has been completed in our laboratory to measure magnetization and magnetostriction at high temperatures simultaneously and accurately, by means of which we are making further researches into this point.

⁽³⁾ S. Kaya, J. Faculty Sci., Hokkaido Imp. Univ., 2 (1938), 29.

⁽⁴⁾ R. M. Bozorth, Phys. Rev., 46 (1934), 232.

Summary

Measurements were made on the effect of the magnetic anneal with wire-form specimens prepared from alloys of nickel and cobalt and the following results were obtained.

- (1) The effect of magnetic anneal on the magnetic permeability is more remarkable in wire-form specimens than in ring-form specimens. The highest value of the initial permeability was 1850 obtained with the 20 percent cobalt alloy cooled in the applied field of 40 Oe, and that of the maximum permeability was 35,080 obtained with the same alloy when cooled in the magnetic field of 2 Oe.
- (2) The effect of cooling in the magnetic field begins at the magnetic transformation point and ends at about 400.
- (3) From a result of measurements of magnetization at high temperatures with the specimens treated in various ways, it is concluded that the effect of the magnetic anneal may be closely connected with plastic flow due to magnetostriction at high temperatures.

The present investigators appreciate the cooperation given by Messrs. Minoru Takahashi and Tatsuo Kôno, with which the present investigations have been carried out.