

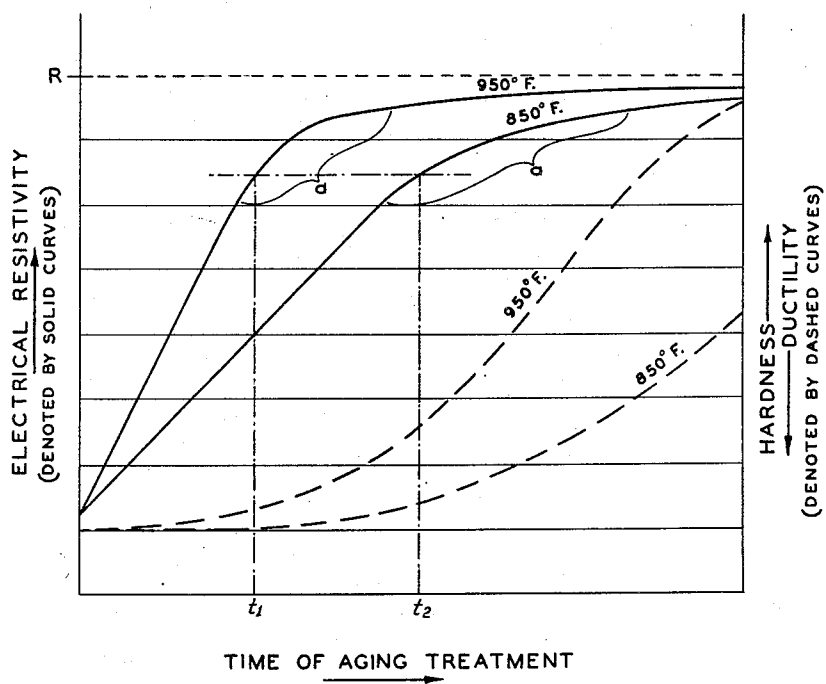
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HEAT TREATMENT OF RESISTOR ALLOYS

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HEAT TREATMENT OF RESISTOR ALLOYS

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This invention relates to alloys composed predominantly of nickel, chromium and manganese, and is directed particularly to the provision of a method for heat treating such alloys so as to increase their electrical resistivity without causing any significant or objectionable decrease in their ductility.

Improved alloys for drawing into wire for use in winding electrical resistors are described in co-pending application Serial No. 130,066 filed November 29, 1949, now Patent 2,628,900 issued February 17, 1953, in the names of myself and Charles T. Greenidge, and in co-pending application Serial No. 230,689, filed June 8, 1951, in the names of myself and Clarence H. Lorig. These improved alloys are composed predominantly of nickel, chromium and manganese. The alloy may consist essentially of these three elements only (as described in application Serial No. 130,066), or it may with advantage contain one or more additional elements such as molybdenum (as in application Serial No. 230,689), or aluminum, copper, vanadium, and iron. The electrical resistance of these alloys is quite high even in the as-cast and cold-worked conditions. It can, however, be made outstandingly high by subjecting the alloy to a solution annealing treatment and then to an aging treatment at an elevated temperature substantially below the annealing temperature. When subjected to this treatment, however, these alloys become precipitation hardened with marked loss of ductility. Such hardening and loss of ductility is a serious disadvantage, because normally, after the alloy has been treated to develop its maximum electrical resistivity, it is desirable to wind it into electrical resistance coils, and otherwise to subject it to plastic and mechanical deformations.

I have discovered that these alloys can be subjected to an annealing and aging treatment which develops nearly the maximum obtainable electrical resistance, without increasing the hardness or decreasing the ductility of the alloy deleteriously, if the duration of the aging treatment is suitably limited. Based on such discovery, the method of this invention for increasing the electrical resistivity of an alloy composed predominantly of nickel, chromium, and manganese without decreasing the ductility thereof comprises subjecting the alloy to a solution annealing treatment by heating it to a temperature in the range from 1500° F. to 2100° F., then subjecting the annealed alloy to an aging treatment by heating it at a temperature in the range from 600° F. to 1200° F. for a period of time between ¼ hour and 100 hours, and terminating said aging treatment at a point in that region of the curve of resistivity versus aging time of the alloy being treated at the aging temperature being used where such curve begins to flatten out. By such treatment, the electrical resistivity of the alloy is increased to a value near the maximum obtainable, without decreasing the ductility of the alloy or increasing its hardness to any objectionable extent.

Among the alloys to which the method of this invention has been applied with success are alloys consisting

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essentially of 50% to 70% nickel, 8% to 28% chromium and 15% to 36% manganese, as described in the aforementioned co-pending application Serial No. 130,066, and alloys consisting essentially of 44% to 70% nickel, 14.0% to 27.2% chromium, 15.9% to 36.4% manganese and 0.1% to 4.0% molybdenum, as described in the aforesaid co-pending application Serial No. 230,689. The method of the invention has also been applied successfully to other alloys composed predominantly of nickel, chromium and manganese, including such alloys which also contain fractional percentages of impurities such as silicon and carbon, and such alloys which contain small percentages of intentional additions of aluminum, copper, vanadium and iron. In defining such alloys as being composed predominantly of nickel, chromium, and manganese, I mean that these three elements will in general account for more than 80% by weight of the alloy, and often will account for 95% or more by weight thereof.

The solution annealing of these alloys is effected by heating at a temperature in the range from 1500° F. to 2100° F., and preferably in the upper portion of this range, that is, from 1700° F. to 2100° F. The time required for the solution annealing treatment depends heavily on the annealing temperature, ranging from as much as 100 hours at a temperature of 1500° F. to as little as two seconds (in the case of fine wires) at a temperature of 2100° F. The time required is, of course, also dependent on the physical size of the article being treated, being much less for small diameter wires than for large diameter bars or rods. It is also to some extent dependent on the prior history of the alloy. For example, if the alloy is in the as-cast condition, the solution treating time must be longer than if the alloy has undergone some measure of mechanical working. Again, a longer time is required to effect solution annealing if the alloy has previously been subjected to an aging treatment with resultant substantial precipitation hardening, than if it has not been previously aged.

Following the solution annealing treatment, the alloy should be cooled quite rapidly to room temperature. Cooling in the open air is quite sufficient for alloys in the form of wire smaller than ¼" in diameter. Shapes of larger section preferably are cooled by quenching in water or oil from the solution annealing temperature. It is possible, though not generally practical, to cool the alloy only to the aging temperature if it is desired to subject it to the aging treatment directly after annealing.

A particular type of solution annealing treatment, designated as strand annealing, can be used with advantage in solution annealing wires in carrying out the method of the invention. This strand annealing step involves passing a wire of the alloy through a long furnace at such rate that the time required for it to traverse the heating zone of the furnace, where it is held at the desired annealing temperature, corresponds to the duration of the solution annealing operation.

The aging treatment is carried out by simply reheating the solution-annealed alloy to a temperature in the range from 600° F. to 1200° F., and holding it at such temperature for a suitably limited period of time between ¼ hour and 100 hours. In most instances, the aging treatment will be carried out at a temperature in the range from 800° F. to 1000° F. for a period of time ranging from ½ hour to 10 hours; and for most satisfactory commercial operations the aging temperature is held within the relatively narrow range from 850° F. to 950° F. and the aging time is in the range from 1 to 5 hours.

It is characteristic of the invention, and of critical importance, to terminate the aging treatment at the proper time. If the time of the aging treatment is too long,

the alloy will become objectionably hardened, with serious loss of ductility; and if it is too short, an undesirably small improvement in electrical resistivity will be effected. The exact duration of the aging treatment will, of course, vary with alloy composition and with the particular aging temperature employed. However, the optimum duration of the aging treatment can readily be determined for any particular alloy by plotting a curve of its electrical resistivity as a function of the time of the aging treatment at the aging temperature selected. When this is done, it will be found that as the duration of the aging treatment is increased, a region of the curve is reached where it flattens out, and beyond this region there is very little increase effected in electrical resistivity of the alloy by prolonging the aging time. If the aging treatment is terminated at a point in this region where the curve begins to flatten out, the extent to which the alloy is precipitation hardened is relatively slight. Hence, by selecting an aging time in this region of the curve, it is possible to produce a ductile wire having an electrical resistivity only slightly lower than the maximum obtainable.

The importance of terminating the aging treatment at the proper time is illustrated by the accompanying drawing, the single figure of which shows curves of electrical resistivity versus aging time and of hardness versus aging time for typical nickel-chromium-manganese alloys at two different aging temperatures (850° F. and 950° F.). The two solid line curves show how the electrical resistivity of the alloy increases quite rapidly during the early stages of the aging treatment, and then begins to flatten off. After passing through a region *a* where this flattening off takes place, the curve becomes approximately horizontal and approaches asymptotically a maximum resistance value *R*. At a relatively high annealing temperature, such as 950° F., the region *a* of the curve is reached at an earlier time in the course of the aging treatment than when a relatively low annealing temperature, such as 850° F., is employed; and also the length of the region *a* along the curve is somewhat shorter at the higher annealing temperature than at the lower. However, the general shape of the curve is the same in both cases.

The manner in which hardness of the alloy increases as the aging treatment continues is shown by the dashed line curves. Very little hardening takes place during the early stages of the aging treatment, but as aging continues the curve of hardness versus aging time bends quite sharply upwardly. This means that substantial hardening of the alloy and substantial loss in its ductility, takes place only after the aging treatment has continued for a substantial period of time.

It will be noted that by terminating the aging treatment at a time corresponding to a point low in the flattening-off section *a* of the curve of resistivity versus aging time, such as at the time *t*₁ for the alloy being aged at the temperature of 950° F. or at the time *t*₂ for the alloy being aged at the temperature of 850° F., it is possible to increase the electrical resistivity of the alloy to a value near the maximum obtainable, and to do so without increasing the hardness or decreasing the ductility of the alloy deleteriously.

Following are examples of the treatment of nickel-chromium-manganese alloys in accordance with the method of the invention, it being understood that the invention is not in any way limited in scope by these examples.

Example I

An alloy composed nominally of 60% nickel, 10% chromium and 30% manganese was melted in an electric furnace and cast in an ingot mold. The cast ingot was forged at 1825° F. to a square bar, and the bar was then hot-rolled at 1800° F. to a rod ¼ inch in diameter. The rod then was drawn cold to a wire 0.070 inch in diameter, and the resulting wire was solution annealed by

heating for 1¼ hours at 1800° F. and then was cooled in the open air to room temperature. The electrical resistivity of the wire in the solution annealed condition was found to be 869 ohms per mil foot. Upon aging the solution annealed wire for 48 hours at 700° F., the resistance was increased markedly to 960 ohms per mil foot without increasing the hardness of the wire or decreasing its ductility to any significant extent. By continuing the aging time at this temperature for a total of 72 hours, the electrical resistivity was increased somewhat further to 990 ohms per mil foot, but the hardness of the alloy was increased sufficiently, and its ductility correspondingly decreased, so that any sharp bending or coiling of the wire, without again annealing it, could not be done. By continuing the aging treatment for a total of 96 hours at 700° F., the resistivity of the alloy was increased a further small amount to 1007 ohms per mil foot, but by doing so the alloy became hardened to such an extent that it was brittle and would break upon any attempt to bend it; hence it could not be subjected to any of the common mechanical deformations in commercial resistor-manufacturing operations.

Example II

An alloy composed of 53.95% nickel, 17.3% chromium, 25.9% manganese, and 2.5% molybdenum, and containing 0.35% silicon as an impurity, was melted and cast into ingot form. The cast ingot was forged at 1775° F. to a square bar ¾ inch on a side, and this bar was then hot-rolled at 1700° F. to a round rod ¼ inch in diameter. The rod was then drawn at room temperature to a wire 0.070 inch in diameter, with intermediate solution annealing treatments, and was finally solution annealed by heating at 1800° F. for ½ hour and then cooling to room temperature in the open air. The electrical resistivity of the wire in the solution annealed condition was found to be 872 ohms per mil foot for one sample and 867 ohms per mil foot for another sample. These samples were then aged by heating at 900° F. for three hours, after which the resistivity values were found to have been increased to 1030 and 1026 ohms per mil foot respectively. This increase in electrical resistance was not accompanied by any objectionable hardening or loss in ductility, for the aged wires could be bent and coiled with ease at room temperature. By continuing the aging treatment for a total time of 5 hours at 900° F., some further small increase in electrical resistivity could be effected, but the hardness of the wire was so much increased and its ductility so much decreased that fabrication of resistor elements by mechanical deformation of the wire became impractical.

I claim:

1. The method of increasing the electrical resistivity of an age-hardenable alloy composed predominantly of nickel, chromium, and manganese and containing those components in amounts of from about 44% to 70% nickel, about 8% to 28% chromium, and about 15% to 36% manganese, without deleteriously decreasing the ductility thereof which comprises subjecting the alloy to a solution annealing treatment by heating it to a temperature in the range from 1500° F. to 2100° F., then subjecting the annealed alloy to an aging treatment by heating it at a temperature in the range from 600° F. to 1200° F. for a period of time between one-quarter hour and one hundred hours, and terminating said aging treatment at a point in that region of the curve of resistivity versus aging time of the alloy being treated at the aging temperature being used where such curve first begins to flatten out, whereby the electrical resistivity of the alloy is increased to a value near the maximum obtainable without deleteriously decreasing the ductility of the alloy.

2. The method of increasing the electrical resistivity of an age-hardenable alloy composed predominantly of nickel, chromium, and manganese and containing those components in amounts of from about 44% to 70%

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nickel, about 8% to 28% chromium, and about 15% to 36% manganese, without deleteriously decreasing the ductility thereof which comprises subjecting the alloy to a solution annealing treatment by heating it to a temperature in the range from 1500° F. to 2100° F., then subjecting the annealed alloy to an aging treatment by heating it at a temperature in the range from 800° F. to 1000° F. for a period of time between one half hour and ten hours, and terminating said aging treatment at a point in that region of the curve of resistivity versus aging time of the alloy being treated at the aging temperature being used where such curve first begins to flatten out, whereby the electrical resistivity of the alloy is increased to a value near the maximum obtainable without deleteriously decreasing the ductility of the alloy.

3. The method of increasing the electrical resistivity of an age-hardenable alloy composed predominantly of nickel, chromium, and manganese and containing those components in amounts of from about 44% to 70% nickel, about 8% to 28% chromium, and about 15% to 36% manganese, without deleteriously decreasing the ductility thereof which comprises subjecting the alloy to a solution annealing treatment by heating it to a temperature in the range from 1700° F. to 2100° F., then subjecting the annealed alloy to an aging treatment by heating it for one to five hours at a temperature in the range from 850° F. to 950° F., and terminating said aging treatment at a point in that region of the curve of resistivity versus aging time of the alloy being treated at the aging temperature being used where such curve first begins to flatten out, whereby the electrical resistivity of the alloy is

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increased to a value near the maximum obtainable without significantly decreasing the ductility of the alloy.

4. The method of increasing the electrical resistivity of an age-hardenable alloy composed predominantly of nickel, chromium, and manganese and containing those components in amounts of from about 44% to 70% nickel, about 8% to 28% chromium, and about 15% to 36% manganese, without deleteriously decreasing the ductility thereof which comprises subjecting the alloy to a solution annealing treatment by heating it to a temperature in the range from 1500° F. to 2100° F., then upon completion of the annealing operation rapidly cooling the alloy to a temperature below 1200° F., then subjecting the alloy to an aging treatment by maintaining it at a temperature in the range from 600° F. to 1200° F. for a period of time between one-quarter hour and one hundred hours, and terminating said aging treatment at a point in that region of the curve of resistivity versus aging time of the alloy being treated at the aging temperature being used where such curve first begins to flatten out, whereby the electrical resistivity of the alloy is increased to a value near the maximum obtainable without deleteriously decreasing the ductility of the alloy.

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