March 20, 1951 H. SCOTT ET AL 2,545,862

PROCESS OF PRODUCING MECHANICAL ELEMENTS

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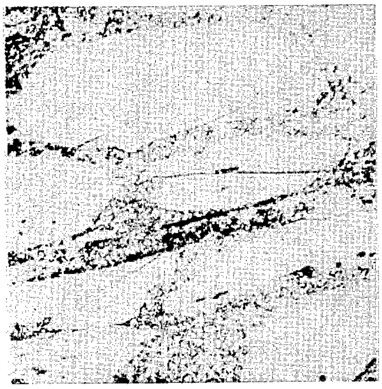

Fig. 1a.

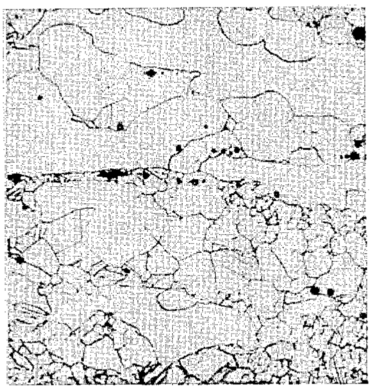

Fig. 1b.

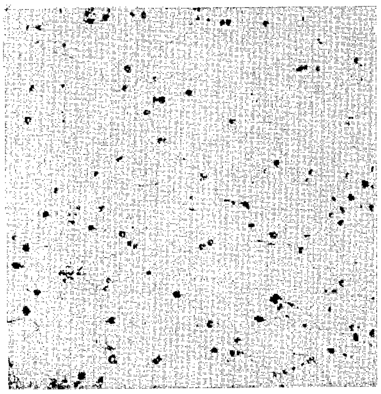

Fig. 2a.

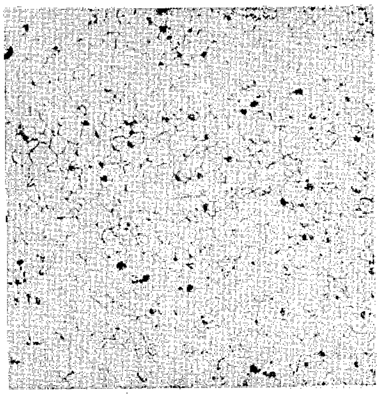

Fig. 2b.

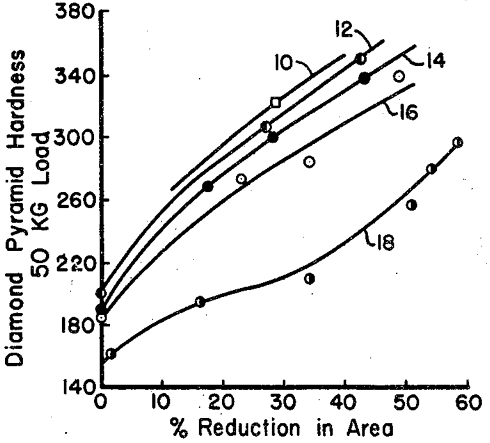

Fig. 3.

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PROCESS OF PRODUCING MECHANICAL ELEMENTS

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5 Claims. (Cl. 148—11.5)

This invention relates to the process of producing mechanical elements from austenitic alloys having good mechanical characteristics at elevated temperatures.

Austenitic alloys employing a precipitation hardening agent have been developed for use in applications at elevated temperatures of 1000° F. to 1600° F. These alloys respond to a precipitation hardening treatment. Such response is retained over a wide range of compositions where the austenitic alloys have chromium and nickel with one or more of the elements iron and cobalt as the base metal thereof, when the precipitation hardening constituent is titanium. These alloys are not critical with respect to the content of the base metal so long as the matrix is a solid solution having only the face centered cubic crystal structure from room temperature up to at least 2200° F. Other elements and impurities may be present in such austenitic alloys in nominal contents, and molybdenum or tungsten in effective contents.

Other alloys of this class are precipitation hardened with molybdenum or molybdenum plus tungsten, and respond to a precipitation hardening heat treatment in the complete absence of titanium, for example, the alloys of U. S. Patent 2,403,128. In this case, however, the hardened content is contingent on the matrix composition in accordance with relations disclosed in that patent. Such alloys, nevertheless, react almost identically with the titanium hardened alloys so far as the teachings of this invention are concerned.

Thus our invention is broadly applicable to austenitic alloys whose matrix is dominantly iron group elements and which may be usefully hardened for service at temperatures of 1000° F. to at least 1600° F. by a combination of a solution treatment which dissolves the hardener and an ageing treatment which induces precipitation hardening.

In producing mechanical elements from such austenitic alloys, it has been found to be quite difficult to produce elements which have consistent mechanical properties. Further, it has not been possible hitherto to work such alloys and so correlate the heat and mechanical treatments as to obtain reproducibly the optimum mechanical properties for a given service application attainable in a specific composition. The alloys considered here are inherently resistant to deformation at high temperatures, being specifically designed to resist creep at service temperatures in the range 1000° F. to 1600° F.

It has been found, furthermore, that a major

improvement in endurance strength and ductility determined at elevated temperatures accrues from refinement of grain size. By the practice of this invention, a fine grain size can be obtained consistently in useful shapes and coarser grain structures can be obtained as well if required for a specific service.

An object of this invention is the provision of a process for producing mechanical elements from precipitation hardenable austenitic alloys having improved mechanical properties at elevated temperatures.

Another object of this invention is to provide for so processing austenitic alloys containing a precipitation hardening constituent that all work hardening resulting from working the alloy to the shape and size of a required mechanical element is removed during the preparation of the element for service.

A further object of the invention is the provision of a process for making mechanical elements from an austenitic alloy in which the alloy is treated to recrystallize the alloy completely to a uniform fine grain size so that the element can be precipitation hardened free from the effects of work hardening and with reproducible mechanical properties.

Another object of this invention is to introduce adequate work hardening into austenitic precipitation hardening alloys by mechanical deformation at temperatures where resistance to deformation is sufficiently low to permit practical forming into finished or semi-finished shapes, so that the alloys can be thereafter completely recrystallized by solution treatment to a fine grain size.

Other objects of this invention will become apparent from the following description when taken in conjunction with the accompanying drawing, in which:

Figures 1*a* and 1*b* are photomicrographs at a magnification of 100 times of alloys within the class defined illustrating the grain size and structure obtained where the alloy is treated in accordance with conventional processes.

Figures 2*a* and 2*b* are photomicrographs at a magnification of 100 times of alloys within the class defined illustrating the refinement of grain size and structure as the alloy is initially treated in accordance with the teachings of this invention, and

Figure 3 is a graph the curves of which illustrate the hardness increase in an alloy as obtained by rolling the alloy at different temperatures in the range of 70° F. to 2200° F.

The alloys to which the process of this invention is applicable may generally be described as precipitation hardenable austenitic alloys having chromium and nickel and at least one of the elements iron and cobalt as the base metal thereof with a precipitation hardening constituent added thereto. Alloys of this type are disclosed and claimed in Patent No. 2,044,165, issued June 16, 1936, to G. P. Halliwell, and consists broadly of 20% to 70% nickel, 60% to 10% cobalt, 5% to 50% iron, .5% to 10% titanium and chromium in amounts ranging from traces up to 20%. Another alloy of this type is that disclosed and claimed in application Serial No. 594,158, filed May 16, 1945, by Scott et al. and now abandoned, consisting broadly of 32% to 42% nickel, 10% to 23% cobalt, 16% to 25% chromium, 3% to 19% iron, 2% to 4% titanium, molybdenum and/or tungsten in an amount to equal the equivalent weight of 2% to 5% molybdenum with minor impurities. Application Serial No. 556,385, filed September 29, 1944, by Scott et al., now Patent No. 2,475,642, also discloses an austenitic alloy of 15% to 35% nickel, 20% to 40% cobalt, 17% to 22% chromium, 3% to 15% molybdenum, 1% to 9% tungsten and the balance iron with minor impurities to which the process of this invention is applicable. Another austenitic alloy of this class is that disclosed in application Serial No. 41,467, filed concurrently herewith, now Patent No. 2,519,406, and comprising from 15% to 35% nickel, 7% to 20% chromium, molybdenum and/or tungsten in an amount to equal from 2% to 5% by weight, 1.3% to 1.9% titanium, not more than 0.4% aluminum and the balance iron with minor impurities. These alloys undergo no phase change between room temperature and the maximum feasible treatment temperature and therefore their grain size can not be refined by heat treatment alone, as is possible with low alloy and plain carbon steels.

In practicing this invention, it has been found to be necessary to apply a controlled work hardening and reheating thereafter of the austenitic precipitation hardenable alloys to break up the crystal structure thereof, and to effect the complete recrystallization thereof whereby when reduced in size a predetermined amount, the alloy can be subjected to a solution treatment at a temperature between 1700° F. and 2300° F. to dissolve the hardening element in the matrix of the alloy, remove the hardness caused by the working, and completely recrystallize the alloy to a substantially uniform predetermined grain size.

The solution treatment selected will naturally depend upon the basic composition of the alloy, the time of holding to be used, and grain size desired to achieve optimum properties for a given application. A grain size at least as fine as ASTM No. 7 is desired for obtaining optimum ductility and endurance strength at moderate service temperatures of 1000° F. to 1400° F., whereas coarser grain sizes yield better rupture and creep properties at higher temperatures. All grain sizes can be obtained by use of the same preparation except for variation in solution temperature and time. Alloys treated in this manner respond readily to an ageing treatment at a temperature of at least 1350° F. to develop the mechanical properties of the alloy.

In working with the austenitic alloys referred to hereinbefore, it has been found that any residual work hardening in the solution treated alloy directly affects the mechanical properties and ageing characteristics of the alloy. This degree of work hardening imparted by prior art forging and forming practice utilizing elevated working temperatures has been difficult to control from piece to piece, and particularly between locations within a given piece of the alloy with the result that inconsistent mechanical properties have been obtained in articles prepared therefrom. Further, it has been found that when the alloys were processed at elevated temperatures in accordance with known processes, that, after working, only partial recrystallization has been obtained at low solution temperatures when attempting to obtain a fine grain size with the result that portions of the original unrecrystallized grains are retained in the alloy in the final article manufactured therefrom. Mechanical elements containing such structures have variable characteristics.

The objectionable microstructures containing mixtures of coarse and fine and recrystallized and unrecrystallized grains in uncontrolled amounts, produced in these alloys as processed in accordance with the prior art are illustrated in Figures 1*a* and 1*b*. The microstructure of Figures 1*a* and 1*b* are those obtained at a magnification of 100 times of alloys having a composition of 40 to 44% Ni, 20 to 24% Co, 11 to 17% Fe, 16 to 20% Cr, 1.8 to 2.4% Ti, 0.4 to 1.0% Mn, 0.3 to 0.8% Si, less than 0.4% Al and less than 0.08% C, Fig. 1*a* being the microstructure obtained when such alloy is rolled from a 1¾ inch square billet from 2100° F. into a 1¼ inch diameter round with a 60% reduction in area. Reheating of such treated stock at 1850° F. for 1 hour failed to effect the complete recrystallization of the alloy to a fine grain size, as clearly shown in Fig. 1*b*.

It has also been found to be impracticable to control the recrystallization of the austenitic alloys in large or complex sections by plastically deforming the alloys at room temperature and then annealing them at a high temperature, such as is done with many other metals in conventional practice. The objection to this procedure is that the initial resistance of the austenitic alloys to room temperature deformation is so great and the increase in hardness with working so rapid that only small reductions may be taken between annealings without increasing considerably the power requirements for working and exceeding the safe limit of stresses allowable in grooved rolls and forging dies. Room temperature working is therefore impractical except as a finishing operation.

We have found, however, that the grain size and recrystallization characteristics of this class of alloys can be completely and effectively controlled by a fabrication procedure comprising the following steps:

1. Heating to an elevated temperature at which the alloys have a relatively low resistance to deformation and at which partial recrystallization occurs during working.
2. Rapid refinement of the grain size by a series of controlled working and reheating cycles.
3. Working to a semi-finished shape.
4. Finish working at as high a temperature as possible, to obtain minimum resistance to deformation, consistent with the requirement that no recrystallization occur during working.
5. Solution treatment to recrystallize the alloy completely to a desired grain size.
6. Ageing for maximum hardness and stability.

Normal forming techniques, such as forging and rolling which are commonly used to produce useful finished or semi-finished shapes, are modified and controlled, as will be described hereinafter, so as to introduce work hardening which can be completely eliminated by a low temperature solution treatment thereby producing a fine and uniform grain size required to obtain optimum and reproducible properties for many applications. Other benefits accruing from the practice of our invention will be apparent from the following description.

Rapid refinement of the grain size of the coarse original structure, in many cases coarser than ASTM No. −10, is achieved by initial application on a cast ingot or coarse grained billet of working either by forging or rolling starting with the alloy heated at a temperature of 2050° F. to 2300° F. In this temperature range the alloys have a relatively low resistance to deformation and least tendency for cracking during working. A distinctive feature of the initial working practice of this invention is the use of a starting temperature such that only partial recrystallization occurs during working and most of that immediately after the first pass on rolling or first sequence of blows on forging. Such recrystallization during working is confined principally to regions around grain boundaries and twin bands. Completion of recrystallization is accomplished by soaking at the starting temperature after reheating for the next reduction cycle. Soaking time at the working temperature prior to the first reduction is governed only by temperature uniformity considerations, but on reheating thereafter it is a major and critical factor in eliminating the condition illustrated by Figs. 1*a* and 1*b* discussed previously and producing homogeneous grain refinement. We have found that by controlled soaking on reheating at the initial forming temperature, the work is completely recrystallized to a grain size several numbers finer than the original. Repeating this operation a still finer grain size is obtained until a No. 2 ASTM grain size or finer is obtained.

An important advantage gained by cyclic working and reheating just long enough for recrystallization to be completed is that more grain refinement can be obtained for a given change in shape or reduction in area than by any working process known in the prior art. Cracking of ingots or billets on forging is thereby minimized, and we find, moreover, that the metal processed according to our invention is amenable to larger subsequent reductions without surface cracking than material rolled by conventional practice, and alligator cracking of ends is less prevalent. Furthermore, guide round rolling, with its more severe deformation than that of hand round rolling, can be practiced to advantage.

In practicing this invention, the working at elevated temperatures can be applied to stock of the austenitic alloy in the form of either a cast ingot or of a billet which has been forged from the ingot which may have a coarse initial grain size large enough to be readily discerned on an etched section by the naked eye. In accordance with this invention, the stock is plastically deformed at the elevated temperature by an amount equivalent to a reduction in area between 5% and 30%, preferably between 8% and 25%, the worked bar being then reheated at substantially the initial heating temperature after each of the reductions has been obtained. Each reduction and reheat constitutes a step in processing the alloy, a plurality of such steps being employed. The purpose of each step is to effect substantially complete recrystallization of the alloy before the following deformation is applied. As will be understood each of the reductions referred to may comprise one or more passes through a set of rolls or blows of the forging hammer. However, the total reduction applied by the plurality of steps is equivalent to a total reduction in area between 30% and 80%.

In practice, the alternate deformation and reheating cycles are applied until complete recrystallization of the metal to a grain size at least as fine as No. 2 ASTM is obtained, it being found that as the grain size becomes finer after each deformation and heating, a given reduction in area is more effective in producing work hardening as measured by recrystallization tendency with the result that less time is required in reheating the bar to effect complete recrystallization after succeeding deformations. Preferably the reheating or soaking of the worked metal is only long enough to assure complete recrystallization since grain growth is very rapid at these reheating temperatures.

In processing ingots of the austenitic alloy, reference may be had to an alloy of the type disclosed in application Serial No. 594,158 referred to hereinbefore and having a specific composition of 37% Ni, 20% Co, 18% Cr, 3% Mo, 4% Ti, 0.2% Al, 0.7% Mn, 0.5% Si and the balance iron. In this alloy the titanium content is so high that it renders the cast ingot having a grain size of No. −5 ASTM unforgeable. In accordance with this invention, a section of the ingot was heated to 2100 F. for 15 minutes after which it was plastically deformed in a series of four reductions and four reheatings by rolling it in an open diamond groove. The alternate rolling and heating produced a bar of austenitic precipitation hardenable alloy that, with a total reduction of only 31%, had a fully recrystallized uniform grain size of No. 6 ASTM. Materials so processed can be readily worked to the shape and size of the mechanical element as described hereinafter.

While it is appreciated that the particular composition of the alloy and the initial cast grain size will influence the size of the first reduction possible and the time necessary to reheat the alloy to obtain complete recrystallization, it is found that in general if the first reduction in area of a cast ingot 2½ inches square or smaller heated to 2100° F. is 8%, the reheating time at the initial heating temperature should be not less than 24 minutes to effect complete recrystallization. If the initial reduction is increased to 12%, then the reheating time can be decreased to 16 minutes. If the initial reduction is 16%, then the reheating time at the initial temperature should be not less than 12 minutes and if the initial reduction is 20%, then the reheating time at the initial temperature should be not less than 9 minutes. In general, the reheating of the alloy is for a period of time between ½ and 60 minutes. On each successive rolling and reheating cycle, the time of reheating can be reduced to about ½ to ⅔ of the previous holding time but for ease of control in manufacturing operations the time in the furnace should not be appreciably less than that required for the billet to attain the approximate temperature of the heating furnace. In most cases, a grain size as fine as No. 2 ASTM can be obtained in three to four reductions and reheatings with a total reduction in area between 30% and 50%. When wrought materials are rolled according to the practice of this invention, the initial grain sizes are much finer than the aforementioned ingot and it has been found that the reheating time after the first reduction may be correspondingly shorter. On the other hand, the cast grain size of a 12 inch square commercial ingot would be much larger than in the smaller ingot, and the initial reheating time after the first reduction should be longer than described above, though succeeding reheatings decrease in the same ratio as before.

As another example of the effect of the alternate working and reheating treatment or cyclic working of this invention, reference may be had to the results obtained in processing a ¾ inch diameter wrought bar of an alloy composed of 25.1% nickel, 13% chromium, 3.01% molybdenum, 2.05% titanium, 0.15% aluminum, 0.69% manganese, 0.65% silicon, and the balance iron. After soaking for one hour at 2100° F. the alloy of this bar had a grain size of No. 3 ASTM as shown in Fig. 2*a*. It was then reduced in area 10% in a hand round groove, reheated at 2100° F. for 2 minutes, reduced 21%, reheated at 2100° F. for 1 minute, and then reduced 15% after which it was again reheated at 2100° F. for 1 minute to effect the recrystallization thereof. In this case the total reduction was 39%. Upon examination, such treatment was found to have completely recrystallized the alloy to a uniform grain size of No. 7.2 ASTM as shown in Fig. 2*b*.

As another example of the grain refinement obtained in following the initial cyclic treatment of this invention, another bar of the same alloy was soaked for ½ hours at 2200° F. at which time it had a grain size of No. 1.8 ASTM and immediately thereafter it was subjected to a 25% reduction in area. After reheating for 1½ minutes at the initial temperature, it was then reduced 16%, reheated for ¾ minute, reduced 17%, and again reheated for ½ minute with the result that the alloy had a uniform grain size of No. 6.2 ASTM. The total reduction in area on rolling from the initial state was 49%.

Likewise another billet of the same alloy which had a grain size of No. 3.4 ASTM when soaked at 2300° F. for 5 minutes was immediately subjected to the alternate working and reheating cyclic treatment consisting of a 25% reduction in area, a reheat at the initial temperature for 1½ minutes, a 16% reduction in area, a reheat for ¾ minute, a 17% reduction in area and a reheat for ½ minute after which the bar was found to be completely recrystallized to a grain size of No. 6.2 ASTM. The total reduction in area of this bar by rolling was 49%.

In the three examples listed above, the reheating times, which represent time in the furnace, were sometimes too short for the bar to attain the furnace temperature. Recrystallization, however, was proceeding during the reheating period and was complete at the end of the time intervals selected. The maximum grain refinement for a given reduction in area is obtained by this procedure, as additional holding permits grain growth. The time of heating to produce complete recrystallization depends not only on the size of the piece, its grain size and the severity of deformation, but also upon the initial soaking temperature, 2050° F. to 2300° F., which is employed in reheating the alloy between each deformation step. Such heating times for all wrought and cast alloys of the austenitic type to which the process of this invention is to be applied range from a minimum of about ½ minute at the upper end of the temperature range given to a maximum of 1 hour at the lower end of the range.

In commercial practice, it is found more convenient to hold in the furnace at least long enough for the material to reach the furnace temperature. By employing working temperatures at the lower end of the recommended range, recrystallization does not proceed as rapidly nor does grain growth become as serious if the bar is left in the furnace longer than the optimum time.

In order to effect the necessary reduction in size from ingot or billet to finished article, much more reduction is usually required than is necessary for grain size control purposes. After the grain size has been reduced to ASTM No. 2 or finer by three or four cycles of controlled working and reheating, as described above, working may be continued in the same temperature range of 2050° F. to 2300° F., to bring the material to substantially the finished size of the articles, the only limit on the amount of work being that of the ability of the fabrication equipment to withstand the stresses and the ability of the material to resist cracking as the temperature of working drops. In order to avoid variations in the amount of recrystallization during mill working, which is not easily duplicated and controlled, it is preferable, however, to finish the mechanical deformation starting at a temperature so low that recrystallization does not occur during working or cooling thereafter.

Using working temperatures between 1550° F. and 1750° F., depending on composition, initial grain size, degree of work hardening, and soaking time, we have found that recrystallization does not occur either during working or reheating between successive passes. This step of the process permits control over the degree and uniformity of work hardening required in preparation for solution treatment and attainment of a fine grain size in the finished product.

We have found unexpectedly however that in the use of starting temperatures within the range 1700° F. to 2000° F., depending on the above-mentioned variables, recrystallization does not occur during mechanical deformation by forging or rolling but will occur and go to completion on reheating with moderate and practical soaking times. In this temperature range moreover the alloys are quite amenable to plastic deformation even in closed die or drop forging. Thus they may be finished to size and shape with successive reheats if necessary at temperatures in this range. Soaking time is controlled to produce complete recrystallization to a fine grain size, the minimum attainable being finer after each reduction. For large pieces which are worked slowly and which heat and cool slowly, temperatures at the lower end of the above range should be used, for example 1700° F. to 1850° F. Small pieces, on the other hand, lose their heat rapidly during rolling or forging and can therefore be heated to higher temperatures for the start of working, namely 1850° F. to 2000° F., without danger of loss of work hardening by recrystallization during working. The time of soaking the alloy at the temperature between 1850° F. and 2000° F. is preferably between 15 minutes and 2 hours.

Referring to Fig. 3, curves **10**, **12**, **14**, **16** and **18** are representative of the effect of rolling temperature on the degree of work hardening as measured by increase in hardness on the alloy identified hereinbefore with respect to Fig. 1, the curves representing the effects of rolling temperatures of 70° F., 1600° F., 1800° F., 1950° F. and 2200° F., respectively. These curves illustrate that work hardening applied to the alloy is quite effective even at 1950° F. Moreover, we find that a given reduction at 1950° F. is practically as effective as that at 70° F., judged by recrystallization temperature, even though hardness is lower by 40 points DPH. Thus sufficient work hardening to achieve positive control of the recrystallization behavior of the alloy can be introduced with a considerably smaller hardness increase by working in the range 1550° F. to 2000° F.

In practice, controlled work hardening without recrystallization may be introduced by either rolling or forging the cyclic treated alloy at a temperature between 1700° F., and 2000° F., to introduce therein an effective reduction in area of 20% to 50%. This work hardening may be accomplished in either one or more reductions depending upon the total reduction required. The upper limit of the amount of working in this step is not critical so long as the region of minimum working of the article has at least a 15% reduction in area or the equivalent degree of imparted thereto. If, however, the final size and shape is such that excessive work hardening is developed as evidenced by resistance to overloading of the mill, the workpiece may be reheated one or more times at the inital temperature and again reduced in area at least 15% after each reheat and at each step until the desired size and shape is reached. It is imperative, however, that the work be soaked sufficiently after being reheated to cause complete recrystallization before any further deformation is applied and that the total reduction after the last reheat be the equivalent of a homogeneous reduction in area of at least 15%.

After the controlled work hardening of at least 15% without recrystallization is imparted to the article, it is subjected to a precipitation hardening treatment consisting of subjecting it to a solution treatment at a temperature between 1700° F. and 2300° F. for a few minutes to several hours and preferably between 5 minutes and 2 hours after which it is subjected to an ageing treatment at a temperature between 1350° F. and 1700° F. for a period of 20 hours. The solution treatment selected and time of soaking thereat is contingent on the alloy composition and final grain size desired in the finished article though the cyclic treatment and the controlled work hardening treatment of the alloy as described hereinbefore are employed to yield in all cases a grain size of No. 4 ASTM or finer with appropriate solution treatment. The range in ageing temperatures is selected to permit attainment of maximum hardness on a 20 hour ageing, the lower temperatures being employed in the case of titanium hardened alloys and the upper temperatures being employed in the case of the molybdenum hardened alloys.

Residual work hardening is also desired in the article prior to the solution treatment to facilitate the making of the article. This will become apparent when it is considered that rolled bars almost always require some straightening. If the alloy recrystallizes during rolling or during cooling from rolling temperatures, straightening may critically strain a portion of the worked piece, and upon subsequent solution treatment, exaggerated grain growth will occur with the result that non-uniform and inferior properties are obtained. However, if residual work hardening equivalent to 15% or more reduction in area is present as taught by this invention, the worked piece can be straightened at room temperature, or rolled or drawn thereat a slight amount to straighten the piece without causing critical grain growth on subsequent solution treatment.

To illustrate the preferred practice of our invention, the following example of processing an alloy comprising 42% Ni, 21% Co, 18% Cr, 2.32% Ti, 0.23% Al and the balance iron plus impurities may be cited: A ¾″ square bar of wrought alloy was heated 1 hour at 2100° F. The bar was then reduced 21%, reheated 5 minutes at the initial heating temperature, reduced 16%, reheated 3 minutes, reduced 17% and reheated 2 minutes and then quenched. At this stage the bar had an ASTM No. 7.6 grain size. The final reheating in this case could be eliminated, if desired, because of the heating for rolling which is applied during the second stage that follows.

For the second stage, that is, controlled work hardening without recrystallization, the bar was then heated ½ hour at 1950° F., establishing a grain size of ASTM No. 7.5 and thereafter rolled without reheating to final size with a reduction in area of 32%. Solution treatment at 1800° F. for 1 hour yielded an ASTM No. 8.5 grain size. The hardness of the alloy after ageing 24 hours at 1350° F. was 318 diamond pyramid hardness.

The solution treatment referred to hereinbefore as being applied to the articles having a controlled work hardening therein functions to cause the solution of the precipitation hardening constituent in the matrix of the alloy, to remove the work hardening and thereby free the articles of the effect of residual work hardening therein, and to effect the complete recrystallization of the alloy to a uniform equiaxed grain size as fine as No. 7 ASTM, if desired. After being soaked at the solution temperature of 1700° F. to 2300° F. from a few minutes to 2 hours depending upon the composition of the article, the work hardening applied thereto prior to the solution treatment and the grain size desired, the article is quenched to room temperature, the preferred quenching mediums being air and oil.

In order to effect the precipitation hardening of the alloy article, the quenched article is then subjected to an ageing treatment at a temperature between 1350° F. and 1700° F. for 20 hours, the temperature being chosen to produce maximum hardness in that time. This high ageing treatment temperature is necessary to develop maximum hardness when the work hardening is completely eliminated by the solution treatment as discussed hereinbefore. The high ageing temperature also increases the resulting maximum service temperature for the article and improves the stability of the alloy forming the article. Other ageing times may be used, 5 to 50 hours for example, with corresponding adjustment of ageing temperature but 20 hours is preferred for practical reasons.

Where desired, a double ageing treatment is given the solution treated article to further improve the stability of the alloy at temperatures within the service temperature range of 1150° F. to 1400° F. in the case of titanium hardened alloys. Thus the first ageing treatment is given at a temperature between 1350° F. and 1400° F. to produce maximum hardness after which the alloy article is slowly cooled to a final ageing temperature of from 50° F. below to 100° F. above the anticipated service temperature.

The process of this invention is applicable to the austenitic alloys of the class referred to, and in particular to such austenitic alloys having a low aluminum content of less than 0.4% as an impurity therein and preferably below 0.3%. When treated in the manner described, a uniform equiaxed grain structure is obtained with the absence of directional characteristics together with a controlled grain size selected to yield the best mechanical properties and stability at elevated temperatures required by the wide variety of loading conditions met in service.

We claim as our invention:

1. The process of producing a mechanical element which is to be subjected to stress at an elevated temperature from stock of an austenitic alloy having chromium and nickel and at least one of the elements selected from the group consisting of iron and cobalt as the base metal thereof and containing a precipitation hardening constituent, the steps comprising, heating the stock to a temperature between 2050° F. and 2300° F. to reduce the resistance of the metal to deformation, deforming the heated stock in a plurality of steps with intermediate heating of the worked stock to substantially the initial heating temperature between the deformation steps for a period of time sufficient to impart a substantially uniform temperature approximating the initial heating temperature to the stock and effect only the complete recrystallization of the metal between said deformation steps, said deformation steps being applied to the completely recrystallized metal immediately upon the effecting of the recrystallization of the metal, the reheating and deformation steps being applied until a substantially uniform grain size as fine as No. 2 ASTM is produced, thereafter plastically deforming the alloy at a temperature between 1550° F. and 2000° F. to form the mechanical element without effecting recrystallization of the alloy, subjecting the element to a temperature above the recrystallization temperature of the plastically deformed element to dissolve the hardening constituent and completely recrystallize the alloy of the element to a substantially uniform grain size as fine as No. 4 ASTM whereby all hardness resulting from deforming the alloy to the element is removed, and thereafter ageing the element at a temperature between 1350° F. and 1700° to effect a substantial increase in hardness.

2. The process of producing a mechanical element which is to be subjected to stress at an elevated temperature from stock of an austenitic alloy having chromium and nickel and at least one of the elements selected from the group consisting of iron and cobalt as the base metal thereof and containing a precipitation hardening constituent, the steps comprising, heating the stock to a temperature between 2050° F. and 2300° F. to reduce the resistance of the alloy to deformation, applying deformation to the heated stock in a plurality of steps each of which effects a deformation in an amount equivalent to a reduction in area between 5% and 30%, reheating the stock between the deformation steps at substantially the initial temperature for a period of time between ½ and 30 minutes, the time of reheating decreasing after each successive deformation to between ½ and ⅔ of the time of the previous reheating, subjecting the deformed stock to a solution treatment comprising heating the deformed stock at a temperature between 1700° F. and 2300° F. for a period of time between 5 minutes and 2 hours to dissolve the hardening constituent and completely recrystallize the alloy whereby all hardness resulting from the deformation of the stock is removed from the deformed stock, quenching the deformed stock from the solution temperature, and ageing the alloy by subjecting it to a temperature between 1350° F. and 1700° F. for a period of time between 5 and 50 hours.

3. The process of producing a mechanical element which is to be subjected to stress at an elevated temperature from stock of an austenitic alloy having chromium and nickel and at least one of the elements selected from the group consisting of iron and cobalt as the base metal thereof and containing a precipitation hardening constituent, the steps comprising, heating the stock at a temperature between 2050° F. and 2300° F. to reduce the resistance of the alloy to deformation, deforming the heated stock an amount equivalent to between 5% and 30% reduction in area, repeating the alternate steps of heating and deforming to effect a total deformation equivalent to a total reduction in area between 30% and 80% and impart a uniform grain size not coarser than No. 2 ASTM thereto, the heating of the alloy between the successive deformation steps of said alternate steps being at substantially the initial temperature for a period of time between ½ and 30 minutes and being progressively reduced to between ½ and ⅔ of the time of the previous heating, reheating the deformed stock to a temperature between 1700° F. and 2000° F. for a period of time between 15 minutes and 2 hours, deforming the heated alloy an amount equivalent to a reduction in area of at least 15% to the desired shape of the mechanical element without effecting recrystallization of the alloy, subjecting the mechanical element to a solution treatment comprising heating the element at a temperature between 1700° F. and 2300° F. for a period of time between 5 minutes and 2 hours to dissolve the hardening constituent and completely recrystallize the alloy of the element to a substantially uniform fine grain size as fine as No. 7 ASTM, quenching the alloy element from the solution temperature, and ageing the alloy element at a temperature between 1350° F. and 1700° F. for a period of time between 5 and 50 hours.

4. The process of producing a mechanical element which is to be subjected to stress at an elevated temperature from stock of an austenitic alloy having chromium and nickel and at least one of the elements selected from the group consisting of iron and cobalt as the base metal thereof and containing a precipitation hardening constituent, the steps comprising, heating the stock at a temperature between 2050° F. and 2300° F. to reduce the resistance of the alloy to deformation, applying deformation to the heated stock in a plurality of steps each of which effects a deformation in an amount equivalent to a reduction in area between 8% and 25%, reheating the alloy between the deformation steps at substantially the initial temperature for a period of time between ½ and 30 minutes, the period of time of the reheating between the deformation steps being progressively reduced to between ½ and ⅔ of the time of the previous reheat, the alternate deformation and reheating effecting the complete recrystallization of the alloy between the deformation steps, deforming the alloy without effecting recrystallization to substantially the shape of the element, subjecting the alloy element to a solution treatment comprising heating the element at a temperature between 1700° F.

and 2300° F. for a period of time between 5 minutes and 2 hours to dissolve the hardening constituent and completely recrystallize the alloy whereby all hardness resulting from the deformation of the alloy to the shape of the element is removed therefrom and the alloy is completely recrystallized to a substantially uniform grain size between No. 6 and No. 9 ASTM, quenching the alloy from the solution temperature, and ageing the alloy by subjecting it to a temperature between 1350° F. and 1700° F. for a period of time between 5 and 50 hours.

5. The process of producing a mechanical element which is to be subjected to stress at an elevated temperature from stock of an austenitic alloy having chromium and nickel and at least one of the elements selected from the group consisting of iron and cobalt as the base metal thereof and containing a precipitation hardening constituent, the steps comprising, heating the stock to a temperature between 2050° F. and 2300° F. to reduce the resistance of the alloy to deformation, applying deformation to the heated stock in a plurality of steps each of which effects a deformation in an amount equivalent to a reduction in area between 5% and 30%, reheating the stock between the deformation steps at substantially the initial temperature for a period of time between ½ and 30 minutes, the time of reheating decreasing after each successive deformation to between ½ and ⅔ of the time of the previous reheating, the alternate deformation and reheating effecting the complete recrystallization of the alloy between the deformation steps, deforming the alloy at a temperature of less than 1750° F. without effecting recrystallization to substantially the shape of the element, subjecting the deformed stock to a solution treatment comprising heating the deformed stock at a temperature between 1700° F. and 2300° F. for a period of time between 5 minutes and 2 hours to dissolve the hardening constituent and completely recrystallize the alloy whereby all hardness resulting from the deformation of the stock is removed from the deformed stock, quenching the deformed stock from the solution temperature, and ageing the alloy by subjecting it to a temperature between 1350° F. and 1700° F. for a period of time between 5 and 50 hours.

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