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3,147,155

HOT-WORKING PROCESS

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3 Claims. (Cl. 148—11.5)

The present invention relates to hot working and, more particularly, to a process for hot working age-hardenable nickel-chromium alloys of the kind that contain aluminum with or without titanium. Such alloys, as is now well known, contain small amounts, for example, up to 0.5%, of carbon, and may also contain additional elements, for example, molybdenum, tungsten, niobium and vanadium, introduced to improve their high-temperature properties. In addition, part of the nickel may be replaced by cobalt, for example, up to 30%, or by iron, for example up to 20%, provided the total content of nickel and cobalt is at least 50%. One or both of the elements boron and zirconium may also be present.

Alloys of this kind are generally not given any special heat treatment before they are hot worked, the work piece merely being heated to the hot-working temperature after any preparation necessary to remove internal or surface defects, for example, cropping or surface machining. The hot-working temperature differs for different alloys, being selected in any particular case to be high enough to ensure that all precipitable phases are in solution but not so high that incipient melting occurs during working as a result of local increases in temperature caused by the working operation. Within the range thus defined, the ease of working generally increases with the temperature, but attempts to work an alloy at either too high or too low a temperature lead to cracking of the work piece. The increase in temperature is greater during hot rolling than during forging so rather lower temperatures must be used for hot rolling than for forging.

It is sometimes possible to hot work an alloy by extrusion at a temperature at which it would crack during hot rolling or forging since the greater constraint of the billet during extrusion inhibits crack formation. The same general considerations as to hot-working temperatures, however, apply to extrusion as to other methods of hot working.

As the total content of titanium and aluminum in the alloys is increased, so is the lowest temperature at which they can be hot worked, and this temperature approaches more and more closely to the incipient melting point. The alloys, therefore, become increasingly difficult to hot work successfully, and even if severe cracking is avoided, the yield becomes very low. Hot working is particularly difficult when the total content of aluminum and titanium exceeds 6% or 7%.

It has now been discovered that by employing a special heat treatment prior to conducting a forging or other hot working operation, nickel-chromium alloys containing at least about 6% aluminum and/or titanium can be readily hot worked.

It is an object of the present invention to provide a novel hot-working process for age-hardenable nickel-chromium alloys.

Another object of the invention is to provide a novel process for hot working nickel-chromium alloys containing at least about 6% titanium and/or aluminum.

Other objects and advantages will become apparent from the following description.

The increase in the temperature needed for hot working is believed to be largely due to the persistence at the grain boundaries, up to increasingly high tempera-

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tures, of an embrittling carbide phase that is not taken into solution in any practical soaking time. This accords with the fact that microstructural investigations of nickel-chromium alloys containing aluminum and titanium have shown that such a carbide phase generally has a higher solubility temperature than the γ' phase, usually represented as $\text{Ni}_3(\text{Ti}, \text{Al})$.

This invention is based on the discovery that in certain nickel-chromium alloys having a high total content of aluminum and titanium the precipitable phase having the higher solubility temperature is the γ' phase, while the carbide phase has a lower solubility temperature. It is further based on the discovery that by means of a suitable heat treatment the formation of a grain-boundary carbide phase in such alloys can be suppressed. For any given alloy the solubility temperatures of the two phases in question may be determined by the usual method of heating samples of the alloy to different temperatures for long enough to reach effective equilibrium, quenching them, and examining their microstructures to ascertain what phases are present.

Generally speaking, the present invention contemplates a novel process of hot working an age-hardenable nickel-chromium alloy that contains aluminum with or without titanium and is such that the solubility temperature of the γ' phase is higher than that of the carbide phase, comprising the steps of solution heating the alloy at a temperature above the solubility temperature of the γ' phase, cooling it to a temperature in the range between the solubility temperature of the γ' phase and that of the carbide phase, maintaining it within this temperature range until a precipitate of γ' phase has formed at the grain boundaries, and then hot working it.

The temperature range between the solubility temperature of the γ' phase and that of the carbide phase will for convenience be referred to hereinafter as the γ' precipitation range. It will, however, be understood that further precipitation of this phase will usually occur at lower temperatures and that at these lower temperatures carbide may also be precipitated.

The purpose of the solution heating is to cause substantially all the precipitable phases to go into solution; and to ensure that this occurs in a reasonable time, the temperature employed is advantageously at least 20° C., and preferably at least 60° C., above the solubility temperature of the γ' phase. To avoid melting, the alloy must, of course, not be heated above its solidus temperature.

In one way of carrying out the invention, the alloy, after the solution heating, is cooled slowly from the solution-heating temperature to a temperature below the γ' solubility temperature at such a rate that its temperature remains within the γ' precipitation range long enough for the desired γ' precipitate to form at the grain boundaries. The slow cooling is conveniently performed by allowing the alloy to cool in the furnace. The rate of cooling preferably does not exceed 4° C. per minute and will generally be slower, e.g., 1° C. to 2° C. per minute. In another way of carrying out the invention, the alloy is cooled at any suitable rate from the solution-heating temperature to a temperature within the γ' precipitation range and is then maintained at that temperature for the necessary length of time. Whichever course is adopted, the length of time during which the temperature of the alloy should remain in the γ' precipitation range will depend on the temperature employed. At temperatures very close to the γ' solubility temperature the rate of formation of the γ' phase will be very slow, becoming faster as the temperature is reduced. In any event the temperature of the alloy preferably remains within the γ' precipitation range for at least 15 minutes.

After the desired grain-boundary precipitate has

formed, the alloy may be allowed to cool to room temperature at any suitable rate, e.g., in air, and subsequently reheated to the hot-working temperature and hot worked. If this is to be done, the slow furnace cooling referred to above may be the most suitable method of causing the γ' precipitate to form. Advantageously, however, the temperature of the alloy is not allowed to fall before the carbide solubility temperature before the hot working; and it is, accordingly, preferred to cause the γ' precipitate to form at a constant temperature as described above and to hot work the alloy substantially at that temperature. Care should, of course, be taken in this case to ensure that the temperature of the work piece is substantially uniform throughout its section before hot working is commenced. It may, therefore, be necessary to maintain the alloy at the temperature in question for longer than is required merely for the formation of the γ' precipitate.

It will be understood that the temperature of the work piece will fall during the hot-working operation and that re-heating to the working temperature will be required from time to time in accordance with ordinary practice. Generally speaking, the hot-working temperature will be with the γ' precipitation range, but some benefit is obtained from the heat treatment when hot-working temperatures below this range are used. The upper limit is imposed by the risk of local incipient melting during working.

The grain-boundary precipitate of the γ' phase is still present during hot working within the γ' precipitation range, and even above the γ' solubility temperature it is only slowly redissolved. Surprisingly, however, this does not impair the hot workability of the alloys. The γ' precipitate at the grain boundaries prevents carbide separating in a harmful form when the temperature falls below the carbide solubility temperature either before the hot working or on cooling between successive stages of hot-working. Any carbide precipitate that does form is largely redissolved on reheating above the carbide solubility temperature. Prolonged heating of the alloy below the carbide solubility temperature may, however, lead to the formation of a carbide precipitate at the grain boundaries and should be avoided. Such a precipitate is also formed if the step of forming the grain-boundary γ' precipitate is omitted, for example, by air cooling the alloy from the solution-heating temperature to below the carbide solubility temperature.

Whatever the mechanism by which formation of a grain-boundary carbide precipitate is suppressed by the heat treatment according to the invention, it is found as a result that the hot working of the alloys is made considerably easier.

As has already been pointed out, it is in general more difficult to hot work a difficultly workable alloy by forging or hot rolling than by extrusion; and it will, therefore, be appreciated that it may be possible to work any given alloy by extrusion without the use of the heat treatment of the invention, whereas successful forging or hot rolling can only be achieved with its use.

It is known that forging and hot rolling may be made somewhat easier if the cast structure of the alloy is first broken down by extrusion, and it is also advantageous to do this in carrying out the present invention.

Nickel-chromium alloys that are difficult to hot work by ordinary procedures but exhibit the special relationship of the γ' and carbide solubility temperatures explained above include those having a total aluminum and titanium content of from about 7.75% to about 9.5% and which contain from about 14% to about 16% chromium, from about 14% to about 25% cobalt, from about 3% to about 5.5% molybdenum, from about 3% to about 4.6% titanium, from about 4% to about 5.4% aluminum, from about 0.01% to about 0.2% carbon, from about 0.01% to about 0.2% zirconium, and from about 0.003% to about 0.1% boron, the balance, apart from impurities, being nickel. The amounts of silicon, manganese and

iron present as impurities should be as low as possible, the silicon and manganese contents not exceeding 0.5% each and the iron content not exceeding 1%. Within this composition range the alloys may, for example, have a chromium content of 14.2% to 15.8%, a cobalt content of 14% to 16%, a molybdenum content of 3.0% to 4.5%, a titanium content of 3% to 4.1%, an aluminum content of 4% to 5.1%, and a zirconium content of 0.02% to 0.1%. A suitable heat treatment for alloys of this latter composition comprises solution heating at 1190° C. for 1.5 hours and cooling to 1000° C. at about 1° C. per minute, after which they may be hot worked at 1120° C.

The improvement in hot workability obtained by means of the invention is illustrated by the following results that were obtained with an alloy (Alloy A) of the composition: carbon 0.17%, silicon 0.31%, iron 0.38%, manganese 0.03%, chromium 14.9%, titanium 3.98%, aluminum 5.07%, cobalt 15.1%, molybdenum 3.39%, boron 0.0085%, zirconium 0.06%, nickel balance. The solubility temperature of the γ' phase in this alloy was found to be 1125° C. and that of the carbide phase 1070° C., and its solidus temperature was 1200° C. Twenty three 1.443-inch diameter rods of this alloy, made by extruding as-cast ingots and machining the resulting extruded bar to size, were solution heated for 1.5 hours at 1190° C., furnace cooled from this temperature to 1000° C. at the rate of 1° C. per minute, and then air cooled to room temperature. Subsequently, these twenty three rods were reheated to 1120° C. and all were hammer forged successfully to 1.125 inch diameter. Twelve similar rods that were merely heated to the forging temperature of 1120° C. without any preliminary heat treatment all ruptured on forging. During the forging operation each of the rods was reheated nine times to the forging temperature, about fifty blows being delivered after each heating.

Round bars of the same alloy were also found to forge down satisfactorily to 0.75 inch square after heating to 1190° C. for 1.5 hours, furnace cooling at the rate of 2° C. per minute to the forging temperature of 1120° C., and holding at that temperature for one-half hour before forging. Similar bars that were only heated to 1120° C. for one-half hour before forging without preliminary heat treatment cracked during forging.

To demonstrate that it is essential to maintain the alloy within the γ' precipitation range for long enough for formation of the γ' phase to occur, an attempt was made to forge extruded round bars of the same alloy from 1.788 inch diameter to 1.25 inch square after solution heat treatment at 1190° C. for 1.5 hours followed by air cooling to room temperature and reheating to the forging temperature of 1120° C. In every case the attempt failed owing to cracking of the bars.

In a further experiment, ingots of an alloy of closely similar composition to Alloy A were extruded to rods which were machined to $3\frac{1}{32}$ inch diameter. The rods were solution heated for 1.5 hours at 1190° C., furnace cooled to 1000° C. at 1° C./minute, and then air cooled to room temperature. Ninety rods were then reheated to 1080° C.-1090° C. for 45 minutes and then hot rolled to $2\frac{5}{32}$ inch diameter (a 35% reduction in area) in 15 passes, with intermediate reheats to 1080° C.-1100° C. when required. One rod split during rolling but the remaining 89 were successfully rolled without cracking.

When the hot working and any further shaping of the alloy to its final form, for example, to rotor blades or discs or other parts of gas-turbine engines, have been completed, the article or part will generally need to be given a further solution and ageing heat treatment to develop the full creep-resisting properties of the alloy.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention, as those skilled in the art will readily understand. Such modifications and variations are considered

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to be within the purview and scope of the invention and appended claims.

I claim:

1. A process of hot working an age-hardenable nickel-chromium alloy having a total aluminum and titanium content from about 7.75% to about 9.5% and which consists essentially of about 14% to about 16% chromium, about 14% to about 25% cobalt, about 3% to about 5.5% molybdenum, about 3% to about 4.6% titanium, about 4% to about 5.4% aluminum, about 0.01% to about 0.2% carbon, about 0.01% to about 0.2% zirconium, and about 0.003% to about 0.1% boron, the balance, apart from impurities, being nickel, said alloy being such that the solubility temperature of the γ' phase is higher than that of the carbide phase, comprising the steps of solution heating the alloy at a temperature above the solubility temperature of the γ' phase, cooling it to a temperature in the range between the solubility temperature of the γ' phase and that of the carbide phase, maintaining it within this temperature range until a precipitate of γ' phase has formed at the grain boundaries, and then hot working it.

2. A process of hot working an age-hardenable nickel-chromium alloy having the composition 0.01% to 0.2% carbon, from 14.2% to 15.8% chromium, from 14% to 16% cobalt, from 3.0% to 4.5% molybdenum, from 3% to 4.1% titanium, from 4% to 5.1% aluminum, the sum of the titanium and aluminum contents being from 7.75%

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to 9.2%, from 0.003% to 0.1% boron, from 0.02% to 0.1% zirconium, the balance, apart from impurities, being nickel, which comprises the steps of solution heating the alloy at 1190° C., cooling it to 1000° C. at the rate of 1° C. per minute, and subsequently hot working it at 1120° C.

3. A process for producing a hot worked, age-hardenable, aluminum-containing, nickel-chromium alloy consisting essentially of about 14% to about 16% chromium, about 14% to about 25% cobalt, about 3% to about 5.5% molybdenum, aluminum with optional additions of titanium such that the total amount of aluminum plus titanium is at least about 6% and up to 9.5%, about 0.01% to about 0.2% carbon, about 0.01% to about 0.2% zirconium, about 0.003% to about 0.1% boron, and the balance, apart from impurities, being nickel, said alloy being such that the solubility temperature of the γ' phase is higher than that of the carbide phase, which comprises the steps of solution heating the alloy at a temperature above the solubility temperature of the γ' phase, cooling it to a temperature in the range between the solubility temperature of the γ' phase and that of the carbide phase, maintaining it within this temperature range until a precipitate of γ' phase is formed at the grain boundaries, and thereafter hot working the said alloy.

No references cited.

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,147,155

September 1, 1964

William S. Lamb

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 59, for "hot working" read -- hot-working --; column 2, line 47, for "couse" read -- course --; column 3, lines 6 and 7, for "tempedature" read -- temperature --; line 7, for "before" read -- below --; line 20, for "re-heating" read -- reheating --; line 23, for "with" read -- within --; same column 3, line 36, for "hot-" read -- hot --; column 4, line 56, for "1190 C." read -- 1190° C. --; column 5, line 7, for "tabout" read -- about --.

Signed and sealed this 27th day of April 1965.

(SEAL)

Attest:

ERNEST W. SWIDER
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