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Moore

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[54] **METAL TREATMENT**

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[58] **Field of Search** 148/125

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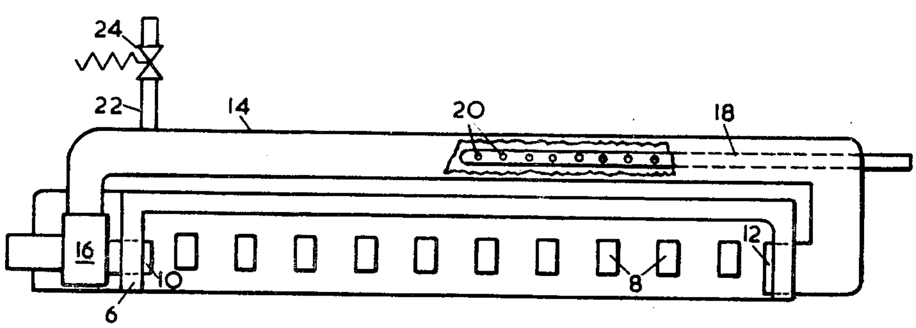
[57] **ABSTRACT**

A metal hardening process including a cooling of the metal to a temperature in the range –80° to –120°C prior to tempering. The cooling is preferably effected in a liquid bath cooled by a liquefied gas.

14 Claims, 3 Drawing Figures



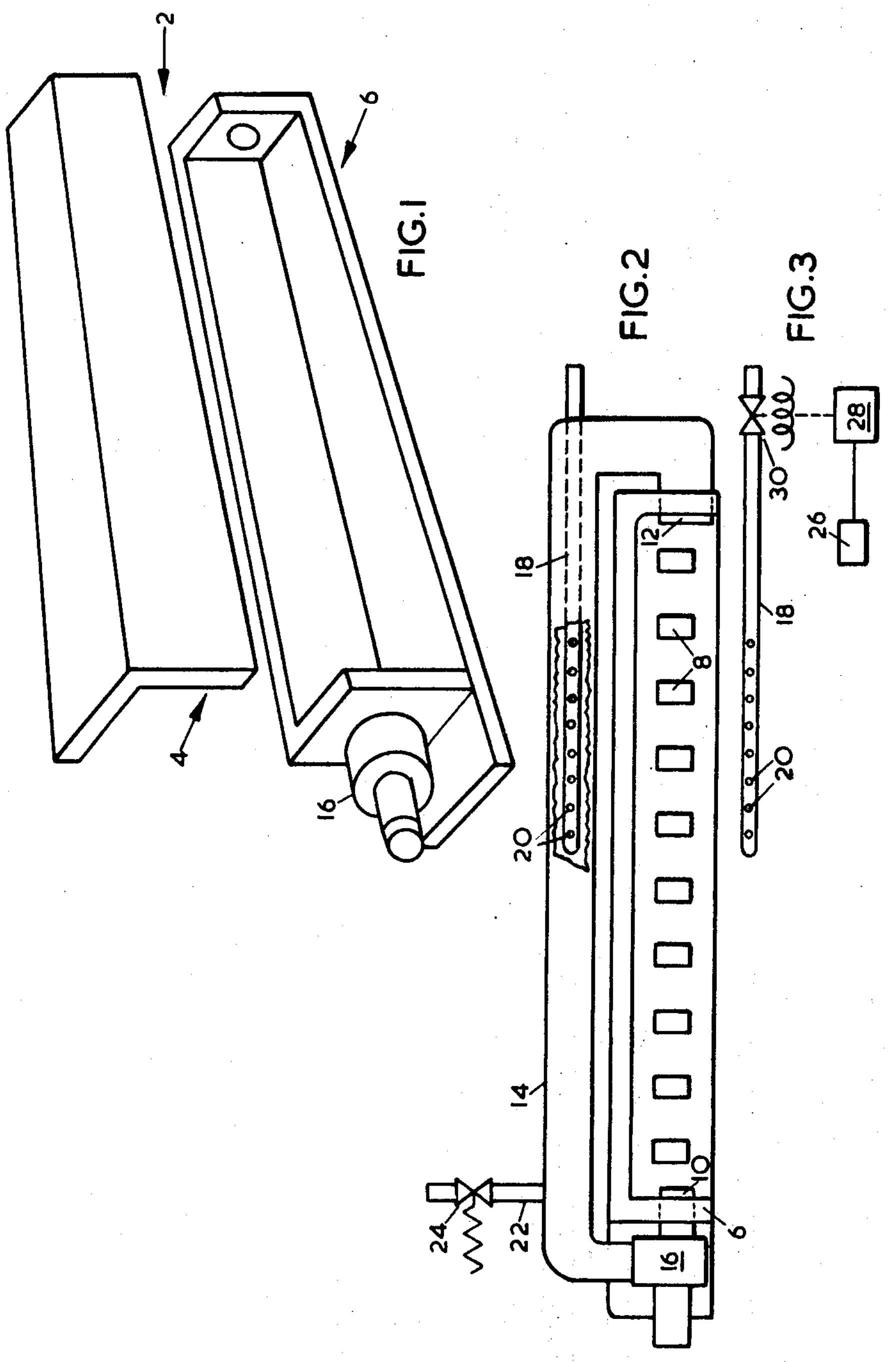


FIG.1

FIG.2

FIG.3

METAL TREATMENT

This invention relates to metal treatment. In particular it relates to an improved process for hardening metals.

Processes for metal hardening generally include several steps of successive heating to elevated temperature and cooling to ambient temperature. Proposals have been made to include also a step in which the metal is cooled to temperatures substantially below 0° C but difficulties have arisen in the methods for effecting this cooling. Direct contact with a cold liquid such as liquid nitrogen imposes severe strains on the metal structure that can only partially be avoided by means of such expedients as wrapping the metal in a protective layer of a material such as paper. Additionally it is believed that known processes have not utilised the optimum temperature range for the sub-zero cooling step, cooling to the temperature of liquid nitrogen (−196° C) being unduly low for most purposes. Another important factor is the stage in the hardening process at which the sub-zero cooling step is performed.

Accordingly the invention provides a metal hardening process in which the metal is, in sequence, heated to an elevated temperature; quenched to a temperature not less than ambient; cooled slowly to a temperature within the range −80 to −120°C by means of liquefied gas in a zone which is initially at or near ambient temperature or by means of cold gas; allowed to return to ambient temperature; tempered at elevated temperature; and cooled to ambient temperature.

The manner in which the initial heating is conducted is chosen according to the metal being treated. For example in the treatment of a tool steel the initial heating is normally effected by preheating to a temperature of the order of 650° to 900°C followed by heating for a short period (say 1 to 5 minutes) to a temperature in the range 750° to 1,325°C.

The quenchant is normally at a temperature initially in the range ambient to 500°C. Gases such as air or nitrogen can be used. Suitable liquid quenchants for use at such temperatures include various oils and molten salts. Any residual oil or salt on the metal should preferably be removed prior to cooling to the temperature in the range −80° to −120°C. If the quenchant is at a temperature above ambient the quenched metal should be allowed to cool to ambient temperature before being transferred to the cooling zone. The period between reaching ambient temperature and introduction into the cooling zone should preferably not exceed 3 hours and preferably is as short as possible so as to minimise stabilisation of austenite.

In the cooling to a temperature within the range −80 to −120°C slow cooling is necessary to ensure that an unduly high temperature difference is not created between adjacent layers of the metal. The metal should preferably be retained at the chosen temperature in the range −80 to −120°C for a period of at least the same duration as that of cooling from ambient to the chosen temperature. In order to return to ambient temperature the metal is then preferably left to stand in air at ambient temperature.

Generally it is preferred that the cooling zone is a liquid bath cooled by liquefied gas. Alternatively the bath can be cooled by cold gas evolved from a liquefied gas.

Suitable liquids for the liquid bath include low melting organic liquids such as acetone.

Suitable liquefied gases for cooling the second bath include liquid nitrogen, liquid oxygen and liquid argon. Liquid nitrogen is generally preferred.

The liquefied gas is preferably contacted indirectly with the bath liquid, for example by passing the liquefied gas through a pipe or coil immersed in the liquid. Alternatively the liquefied gas can be introduced directly into the liquid, preferably accompanied by agitation of the liquid. Slow cooling is necessary to ensure that an unduly high temperature difference is not created between adjacent layers of the metal. Thus the rate at which the second liquid bath should be cooled is dependent upon the thickness and thermal conductivity of the metal being treated. For an iron ingot of 1cm thickness the bath is preferably cooled from 15°C to −110°C over a period of about 10 minutes.

In certain circumstances however, it is preferred to contact the metal directly with cold gas. For example if the process of the invention is used to treat a large metal workpiece, difficulties can arise in loading the workpiece into a liquid bath. The cold gas is preferably formed by evaporating liquefied gas. Nitrogen is preferred.

To facilitate handling of a large workpiece, the gas cooling zone is preferably in a cold box which can be loaded with the workpiece from the front.

If a cold gas is employed it is desirably introduced in such a manner as to give good contact with the surface of the metal being cooled. The temperature of the gas and the rate at which it is introduced into the cooling zone can be readily selected to give a suitable rate of cooling.

The tempering is normally conducted at a temperature within the range 150° to 600°C for a period of one-half to 2 hours, the particular temperature and period being selected according to the composition and cross-section of the workpiece. In contrast with previous hardening treatments, the process of the invention gives in a single tempering stage a degree of hardness at least equal to that formerly achieved with several tempering stages.

The process of the invention is well suited to the hardening of precipitation hardening alloys such as those of magnesium or aluminium, and is particularly useful for the hardening of steels, especially for tool steels. When using the process of the invention to treat steel, the steps of the procedure following the initial heating at elevated temperature have the effect of reducing the proportion of austenite and improving the nature of precipitated alloy carbides in the steel.

A temperature below −80°C promotes break-down of the austenite molecules, even through the rate of breakdown decreases with decreasing temperatures, but substantially halts the precipitation of alloy carbides. Thus on warming to ambient temperature and in subsequent tempering alloy carbides are precipitated into a steel having a very low austenite content and as a result give a product of improved hardness.

A temperature below −120°C is to be avoided. It may cause undue stress in the metal structure and further, practical difficulty is encountered in finding for the liquid bath a material that remains in the liquid state at temperatures below −120°C.

The process of the invention is advantageous in that it is quicker and requires less energy than a procedure that requires more than one tempering step. Moreover it gives a product that is more resistant to flaking than a product obtained by a process including a conventional cold treatment.

The invention includes within its scope metal that has been treated by the process of the invention.

The following Examples illustrate the process of the invention.

Example 1

Samples of tool steel having the composition, by weight, of 18% tungsten, 4 percent chromium and 1 percent vanadium were hardened according to (a) a conventional treatment and (b) the process of the invention. The samples each measured 2 × ½ × ½'' overall, had the tool angles (ISO) 0, 15, 10, 5, 15, 20 and a noze radius of 0.015 inches.

The tools treated by the conventional method were preheated at 850°C, hardened at 1,280°C for two minutes and quenched in a hot oil bath at 250°C. They were then cooled in air to 18°C and cleaned to remove residual oil. Finally they were twice subjected to a tempering at 550°C for one hour, in each case followed by cooling in air. The tools treated by the method of the invention were also preheated at 850°C, hardened at 1,280°C for 2 minutes, quenched in a hot oil bath at 250°C and cooled in air for 30 minutes. They were then cleaned to remove residual oil and inserted in an acetone bath at 18°C. The acetone bath was provided with a coil through which liquid nitrogen was passed slowly so as to cool the acetone to −100°C in 10 minutes. The acetone temperature was maintained at −100°C for a further 10 minutes by passing a very small amount of liquid nitrogen through the coil to to balance the warming effect of the surroundings and the tools were then removed and allowed to stand in air for 30 minutes to return to ambient temperature (18°C). They were finally subjected to a single tempering at 550°C for 1 hour and air cooled.

A comparative test was then effected using the method and apparatus of RA Billet's UK Pat. No. 1,190,072 A bar of free cutting EN1A steel was placed on a lathe and cut by the different tools in turn. The depth of cut was 0.050 inch and the bar was fed to the tool at a rate of 0.004 inch per revolution. The flank wear of the tools over a specified period was then measured and the results are shown in the table.

Treatment	Length of test (seconds)	Cutting speed (ft/min)	Flank wear (in × 10^{-3})
(a)	2550	320	11
(b)	2850	320	9
(a)	3000	240	7.5
(b)	3000	240	5.5

Example 2

The apparatus in which the cooling to a temperature in the range −80°C to −120°C was effected for this Example is now described, by way of example, with reference to the accompanying drawings, of which:

FIG. 1 is a schematic perspective view of a cold box;

FIG. 2 is a plan view of the cooling apparatus including the cold box shown in FIG. 1; and

FIG. 3 is a circuit diagram of a liquefied gas supply control system which may be used in conjunction with the apparatus shown in FIG. 2.

Referring to FIGS. 1 and 2, a cold box 2 is formed of front and rear sections 4 and 6. The interior of the cold box 2 constitutes a cooling zone in which metal being treated by the process of the invention may be cooled to a temperature in the range of −80° to −120°C. Within the interior of the cold box 2 are located several battens 8 upon which work to be cooled can be placed. The cold box 2 has an inlet 10 and an outlet 12 for vaporised liquid gas. An insulated duct 14 enables gas leaving the outlet 12 to be recirculated to the inlet 10, the recirculation being effected by a blower 16 located just upstream of the inlet 10.

A pipe 18 leads from a source of liquefied gas (not shown) through the wall of the duct 14 to a sprayer 20 located within the duct 14. In operation the evaporated gas in the duct vaporises the liquid spray in carrying it towards the blower 16. Thus a stream of cold evaporated gas is passed over the workpiece on the batten 8.

To prevent an undesirable pressure being created within the cold box 2, a vent pipe 22 leads from the duct 14 and has located therealong a pressure relief valve 24 which may be set to open at a chosen pressure.

In FIG. 3 is illustrated a system which may be used to control the introduction of liquefied gas through the sprayer 20. A temperature sensing element 26 which may be located at the outlet end of the cold box 8 is in circuit with a thermostat 28. The thermostat 28 may be set such that a solenoid valve 30 controlling the liquid supply to the sprayer 20 remains open until the temperature sensed by the element 26 reaches a chosen minimum.

The cold box is constructed from an outer wooden shell and an inner insulating shell of vermiculite which is protected from the cold gas by a lining of aluminium sheet. The internal dimensions of the cold box 2 are 12 by 1 by 1ft.

In this example the workpiece to be cooled is a 10 foot long steel blade. When the quenching has been completed, the blade is transferred ready for location in the cold box 2, the lid (front section 4) of the box 2 removed, the blade placed in position within the box 2 and the lid 4 replaced. Passage of liquid nitrogen into the sprayer 20 is then commenced. The thermostat 28 is set so as to effect the cooling in a number of stages. This is to prevent excessively fast cooling. After the blade is cooled to the desired temperature, it is allowed to return to ambient temperature, tempered at elevated temperature and cooled to ambient temperature.

I claim:

1. A metal hardening process in which the metal is, in sequence, heated to an elevated temperature; quenched to a temperature not less than ambient; cooled slowly to a temperature within the range −80° to −120°C in a region which is initially at or near to ambient temperature by direct contact with cold gas or by indirect contact with cold gas or cold liquid so as to ensure that an unduly high temperature difference is not created between adjacent layers of the metal; allowed to return to ambient temperature; tempered at elevated temperature and cooled to ambient temperature.

2. A process according to claim 1, in which in the treatment of a tool steel the initial heating is effected

by preheating to a temperature of the order of 650° to 900°C followed by heating for a short period to a temperature in the range of 750° to 1325°C.

3. A process according to claim **1,** in which the quenchant is initially at a temperature in the range ambient to 500°C.

4. A process according to claim **1,** in which the quenched metal is allowed to cool to ambient temperature before being transferred to the slow cooling stage.

5. A process according to claim **4,** in which the period between the metal reaching ambient temperature and being subjected to the slow cooling is less than three hours.

6. A process according to claim **1,** in which the metal is retained at the chosen temperature in the range −80° to −120°C for a period of at least the same duration as that of cooling from ambient to the chosen temperature.

7. A process according to claim **1,** in which after the slow cooling stage, the metal is left to stand in air at ambient temperature.

8. A process according to claim **1,** in which the slow cooling is conducted in a liquid bath cooled by liquefied gas.

9. A process according to claim **1,** in which the slow cooling is conducted in a liquid bath cooled by cold gas.

10. A process according to claim **8,** in which the liquefied gas is contacted indirectly with the bath liquid.

11. A process according to claim **8,** in which the liquefied gas is liquid nitrogen.

12. A process according to claim **1,** in which the metal is contacted directly with a cold gas.

13. A process according to claim **12,** in which the cold gas is nitrogen.

14. A process according to claim **1,** in which the tempering is conducted at a temperature within the range 150° to 600°C for a period of one-half to 2 hours.

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