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Backman et al.

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[54] METHOD OF MAKING STAINLESS STEEL

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[52] U.S. Cl. .... 148/12 E; 148/12.4

[51] Int. Cl.<sup>2</sup> .... C21D 7/02

[58] Field of Search ..... 148/12 E, 12.4; 75/128 C, 75/128 W, 128 A

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[57] ABSTRACT

Spring steel (in wire or strip form) is produced from an austenitic stainless steel, the chromium content of which is sufficient to give the steel a metastable austenitic microstructure, by (1) annealing and (2) quenching under conditions to give it a wholly austenitic condition, followed by (3) a very substantial cold reduction which transforms its structure to an at least partially martensitic structure and produces increased hardness.

3 Claims, 4 Drawing Figures

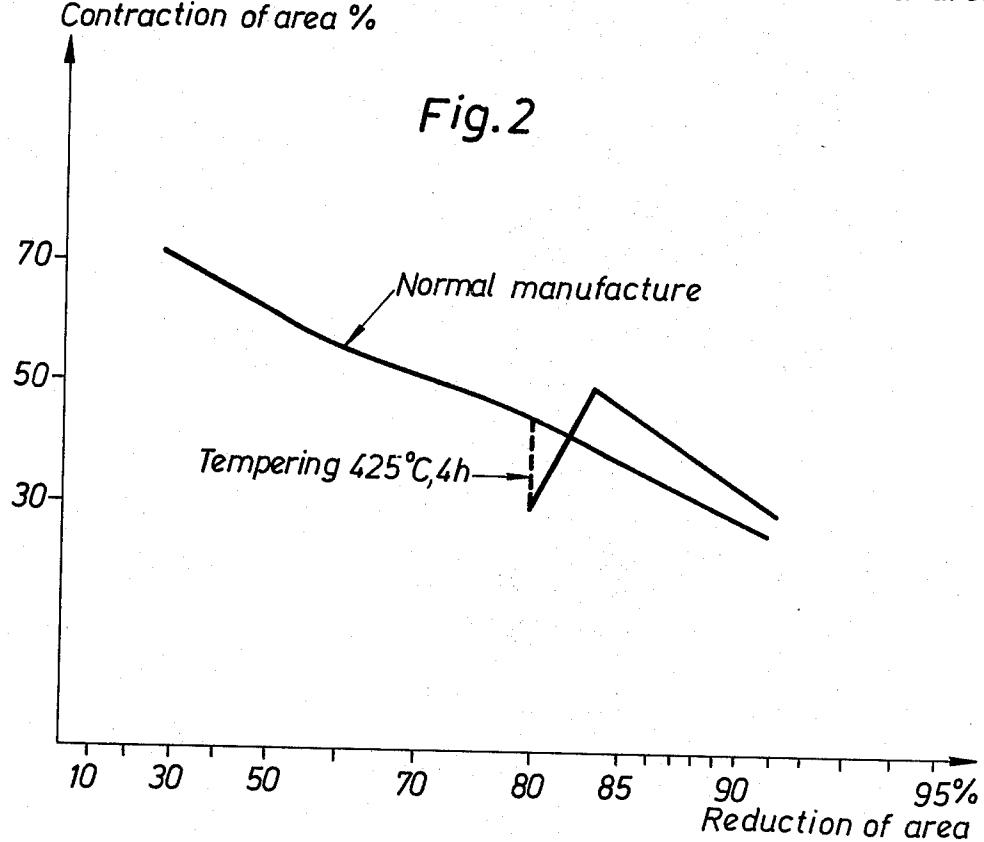
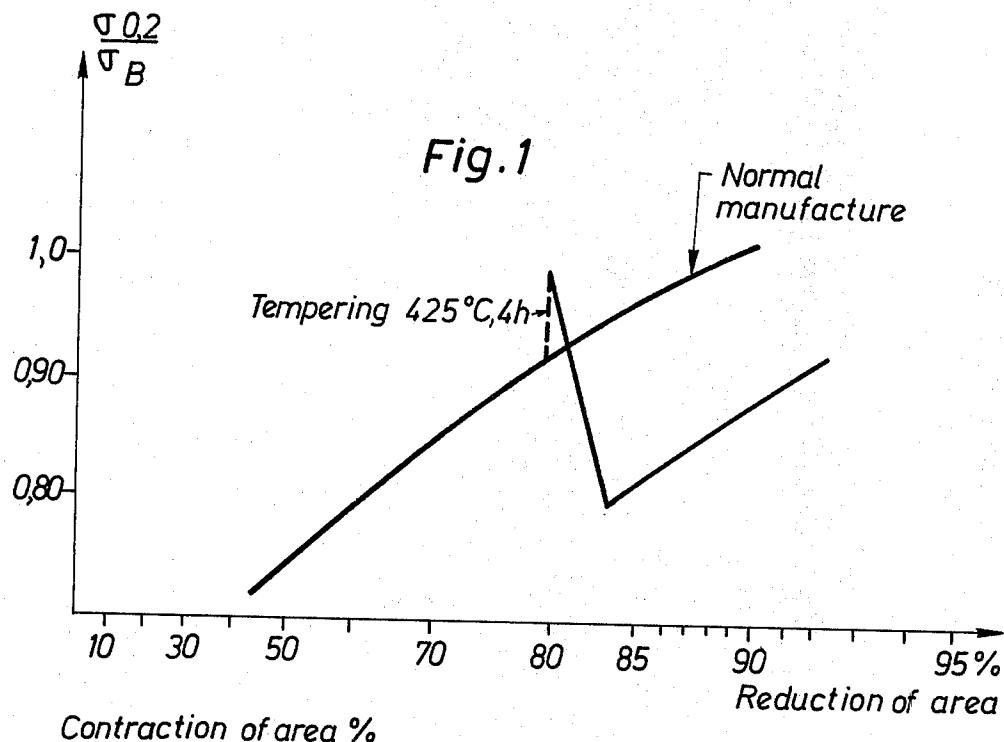


Fig. 3

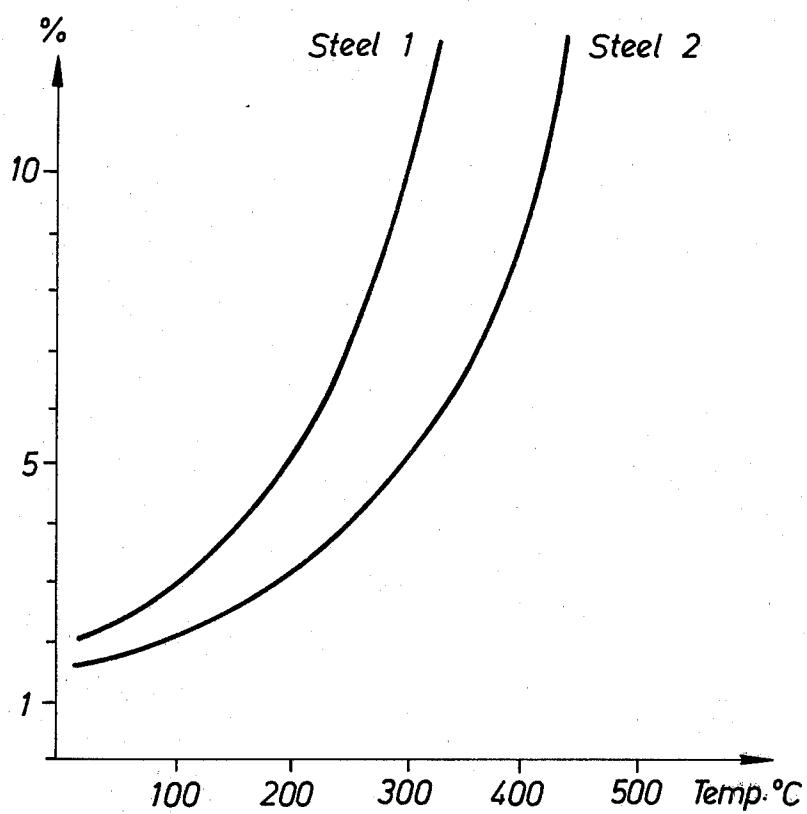
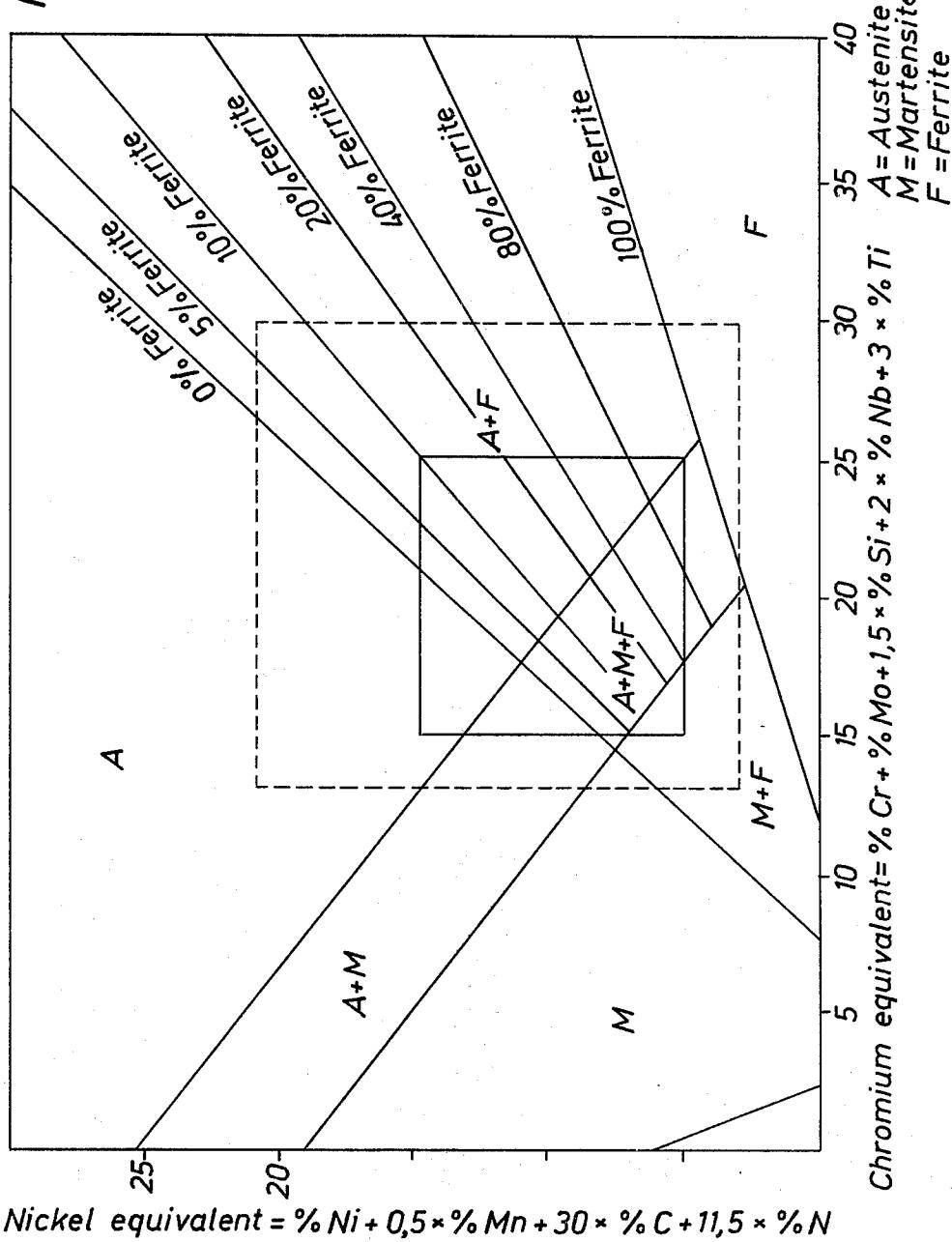


Fig. 4



## METHOD OF MAKING STAINLESS STEEL

The present invention relates to a stainless chromium-nickel steel with high strength, and which at the same time has good ductility. This invention also relates to a new method of treating such stainless steel in the form of band or wire to achieve these new characteristics of stainless steel, said steel then being adaptable for use as a spring material in the form of wire or strip.

High strength material which at the same time has a good ductility is increasingly called for. One method of strengthening steel consists in subjecting the steel to an appreciable cold reduction. This method is especially adaptable for certain types of austenitic stainless steel in which an austenitic structure is partially transformed by cold deformation into a hard martensitic structure. The mechanism of this structural transformation is well known and is disclosed in, for instance, British Pat. Nos. 722,427 and 766,971. These unstable austenitic steels have been in wide use especially as spring materials, and are available in the form of round wire or bands.

The requirement for good ductility often implies that it must be possible to carry out a succession of deformations of such material without any ensuing fractural formations. If the material is in the form of wire there is a requirement that it must be possible to have such wire wound around a bar the diameter of which is approximately the same as that of the wire itself. The normal procedure of processing such steel is to carry out a cold reduction to a level of strength and ductility such that the material then is able to be subjected to a shaping procedure into a final product. Such a product is often also subjected to a final heat treatment at a temperature of 200°-550°C. during a few hours for strengthening purposes.

In order to achieve a sufficiently high ductility with these high strength steels, however, it has been necessary to accept a lower non-optimal strength level. This has been so because the achievement of an increased strength is — in principle — accompanied by a decrease in ductility. In extreme cases a high strength might be attainable but then the material also exhibited such an embrittlement that fracture occurred at minor deformations thereof. This is an effect that closely related with a blocking of dislocations that occurs in martensite as well as in austenite phases in deformation hardening. If the reduction is too high further dislocation movements are impossible due to the mutual blocking thereof. This implies that an additional reduction of said material would result in micro-cracking, which results in an appreciable decrease in ductility.

According to the present invention it surprisingly has now been found that it is possible to avoid the above related lessening of ductility. The invention thus resides in a new stainless steel which exhibits the unique combination of high strength and ductility and which also exhibits good properties under elevated temperature conditions. The articles of the invention also have good resistance to corrosion and to oxidation at the working temperatures.

It is an object of the invention to provide a stainless chromium-nickel steel that is hardenable by cold-work into an austenitic-martensitic microstructure, said steel in the cold-worked condition having a tensile strength in excess of 250,000 p.s.i., but not in excess of 400,000

p.s.i. and preferably not in excess of 390,000 p.s.i., and a high ductility which, measured as contraction of area immediately before rupture, is in excess of 45% and which in addition also exhibits good properties under elevated temperature conditions in terms of relaxation permanence. In its broader aspects, the invention provides such an alloy having a composition consisting essentially of, by weight, about 0.01 to 0.20% carbon, up to about 5% silicon, up to about 10% manganese, from about 13 to about 20% chromium, about 3 to 10% nickel, up to about 2.5% molybdenum, up to about 2.5% aluminum, and the balance being essentially iron except for small amounts of other elements which do not adversely affect the desired properties of the alloy.

It is another inventive object to provide a method for processing an austenitic stainless chromium-nickel steel to high strength and ductility levels. Briefly, such steel is subjected to deformation hardening as a result of cold-deformation through cold-working at large reduction of area after which it is annealed at a temperature between 200° and 550°C. Subsequently, this steel is subjected to a moderate cold-working in which the cross-sectional area is reduced by between 5 and 40%, preferably between 10 and 30%. Due to this method of processing, a deformation hardening occurs such that a considerable gain in ductility is achieved whilst retaining a high tensile strength.

According to the invention, I start with an austenitic stainless steel in the form of wire or strip, this steel having been hot rolled in the usual manner and which has a composition comprising chromium in amounts sufficient to give the steel a metastable austenitic microstructure. After said hot rolling the steel is (or, may be) subjected to a conventional surface treatment such as pickling, grinding, sandblasting or similar treatment. Such a material is then transformed into a wholly austenitic condition by annealing at a temperature of 950°-1100°C. and then quenching in water. Subsequently the steel is subjected to a cold reduction in one or several steps without intermediate annealings at a very large reduction of area, thus partially transforming the austenitic structure into a martensitic structure, an increased hardness simultaneously being obtained.

It heretofore had been known to cold-reduce an austenitic steel in a series of cold deformations with high reductions upon quenching same, thus effecting a so-called deformation hardening of the material. However, it was not derivable from prior knowledge that such a material subjected to a cold deformation next to entering the brittle state if subjected to a subsequent tempering should have a considerable margin of ductility such that even an increase thereof could be gained when carrying out further deformation. By choosing an optimum of reductions and tempering time-temperature relations in such method of processing unexpected combinations of steel characteristics have been found to be achievable. It has thus been possible to achieve a considerable gain in ductility yet retaining or even also increasing the tensile strength level.

FIG. 1 is a diagrammatic showing of the effect of total reduction on the ratio between yield point and ultimate tensile strength;

FIG. 2 shows the effect of total reduction on the contraction of area immediately before rupture;

FIG. 3 diagrammatically represents the improvement in properties under elevated temperature conditions in the case of a steel of the 18-8 type; and

FIG. 4 is a modified Schaeffler diagram showing microstructures, attainable through practice of the present invention, in the cases of selected chromiumnickel steels.

The starting material, treated as related hereinbefore, is upon quenching subjected to cold deformation with high reduction of area next to entering the brittle state, said area reduction amounting to 40-90%, preferably 60-85% thus partially transforming the austenitic structure into martensite — the amount of which being 30-90%, usually 45-85% — while the remainder is austenite with small amounts of ferrite. Subsequently, the material is subjected to tempering at a temperature of 200°-550°C., preferably 250°-450°C., for a suitable time which, according to several factors such as the tempering temperature and the dimensions of the objects may be from some minutes up to 10-12 hours or even longer. The tempering time usually is between 15 minutes and 10 hours, and preferably between 2 and 5 hours. This tempering is necessary so as to relieve those stresses which appear to a locally high degree in the microstructure as a result of the cold working. After said treatment the material is further cold reduced to a moderate reduction of area amounting to 5-40%, preferably 10-30%. The last reduction of area must amount to at least some 5-10% so as to achieve any increased ductility whereas a reduction of

$\sigma_{0.2}$  indicating stress applied to effect a plastic deformation of 0.2%. FIG. 1 illustrates the effect of total reduction on the ratio

$$\frac{\sigma_{0.2}}{\sigma_B}$$

and in FIG. 2 there is illustrated the effect of total reduction on the contraction of area immediately before rupture. In both figures there are also curves illustrating the common method of processing not within the scope of the invention in comparison with those illustrating the method of the invention. For purposes of comparison, steel alloys were prepared and test specimens were made therefrom for carrying out tests the results of which are set forth in the Table following hereinafter. The material made subject of these tests was stainless steel wire subjected to cold drawing upon quench-annealing from 1050° C., the analysis of which was as follows:

C	Si	Mn	Cr	Ni	Mo	Fe
0.09	1.15	1.25	17	8	0.7	bal.

TABLE

Treatment	Dimension mm	Yield point $\sigma_{0.2}$ kp/mm <sup>2</sup>	Ultimate strength $\sigma_u$ kp/mm <sup>2</sup>	Ultimate elongation $\delta_{50\%}$	Contraction of area before rupture %
Reduction 83%	φ1.0	220	230	1.8	40
Reduction 80% + tempering 425°C 4 h	φ1.0	230	235	1.4	30
Reduction 80% + tempering 425°C 4 h + reduction 20%	φ1.0	220	240	3.5	50
Reduction 80% + tempering 425°C, 4 h + reduction 20% + tempering 425°C, 4 h	φ1.0	230	255	2.5	40

area in excess of about 40% will cause a decrease in ductility. This circumstance will be apparent from the diagrams illustrated in the appended drawings. The steel material may be subjected to another tempering after said last moderate cold reduction, the temperature of which final step then being 300°-550°C., preferably 300°-500°C. for a time of 2-5 hours, an increased tensile strength being obtained as a result thereof.

Various measures are used so as to indicate the ductility of a material, such as: ultimate elongation, contraction of area before rupture, i.e. the percentual decrease of area at the place of rupture; and the ratio between yield point and ultimate tensile strength,

$$\frac{\sigma_{0.2}}{\sigma_B}$$

It is apparent from the data in this Table that all three measures of ductility have been improved by practicing the treatment of the invention, i.e. contraction of area before rupture, ultimate elongation and the ratio

$$\frac{\sigma_{0.2}}{\sigma_B}$$

the last moderate cold reduction having been effected upon tempering at about 425° C. The ultimate elongation is measured as  $\delta_{50}$ , i.e. percentual elongation of a wire of 50 mm length as indicated between two points thereon, said last measure being 3.5% after tempering at 425° C. for a period of 4 hours and subsequent cold reduction at 20% reduction of area. It is also apparent from the Table that the common method of processing resulted in a considerably lower ultimate elongation, i.e., a lower ductility.

The Table is also illustrative of the improved ultimate strength reached with the alloy of the invention, which

amounts to 240 kp/mm<sup>2</sup> (343,000 p.s.i.) upon tempering at 425° C. for a time of 4 hours plus 20% reduction of area. After another tempering at 425° C. for a period of 4 hours the strength reached a level of 255 kp/mm<sup>2</sup> (364,000 p.s.i.) whilst the material retained a high ductility, 2.5% in terms of ultimate elongation. The results of these tests thus established that it is possible to achieve a considerable increase of ductility yet retaining a high strength level by carrying out a method of processing according to the invention.

In addition, a considerable gain in the properties under elevated temperature conditions appeared as a result of the cold-work processing related above. This improvement of steel characteristics is illustrated in FIG. 3 and refers to the relaxation permanence, which expression means the percentual loss of applied load at a certain temperature and for a length of time. The diagram of FIG. 3 shows such a curve of steel "1" of 18-8 type not cold-worked according to the invention in comparison with steel "2" which is within the scope of the invention. The curves refer to the percentual loss of an applied load of 60 kp/mm<sup>2</sup> for a period of 24 hours at differing temperatures. The diagram clearly shows the very good relaxation permanence of steel 2, this being a very important property of steels adapted for use as spring material.

Thorough studies of microstructure and steel characteristics have shown that the improvement of ductility thus reached is closely related with changes that occur in dislocation structure by reason of the tempering within the range earlier set forth. Measurements of micro-stresses of cold-reduced and tempered wire of the steel analysis earlier referred to have indicated a drastic decrease of said micro-stresses — primarily in martensite — after said tempering. This implies that stress-relieving occurs along with a lessened risk of fracture during a subsequent deformation. By avoiding occurrence of interior fracture formations in cold reduction, a further increase of dislocation denseness is possible, thus enabling an increased ductility to be obtained.

The effect thus achievable by the method of the invention applies to all those austenitic, or essentially austenitic, steels wherein austenite is partially transformed into martensite as a result of deformation and where a heat treatment gives rise to precipitations which give rise to an increased strength level. The chemical analysis thus must comprise an amount of chromium, usually in excess of 13%, that is sufficient to provide a metastable austenitic microstructure under the conditions that apply while deforming the steel. The method of the invention is applicable to steel alloys having a composition in weight percentages, consisting essentially of about 0.01 to 0.20% carbon, up to about

5% silicon, up to about 10% manganese, about 13 to 20% chromium, about 3 to 10% nickel, up to about 2.5% molybdenum, up to about 2.5% aluminum, and the balance being essentially iron except for small amounts of other elements which do not adversely affect the desired properties of the alloy.

In FIG. 4 there is shown a modified Schaeffler diagram from which is derivable the microstructure that will be the result of a certain steel analysis. The dotted 10 rectangle indicates the area within which the alloys appear in the broader aspects of the invention, whereas the smaller square therein corresponds to the more narrow and preferred ranges of analysis according to the practices of my invention, this last squared area being limited by the lines set by chromium equivalents in amounts of 15-25% and nickel equivalents in amounts of 5-15%, said chromium equivalent being given by the relation (%Cr + %Mo + 1.5 × %Si + 2 × %Nb + 3 × %Ti) and said nickel equivalent being given by the relation (%Ni + 0.5 × %Mn + 30 × %C + 11.5 × %N).

We claim:

1. A method of making austenitic stainless steels having high tensile strength in excess of 250,000 p.s.i. but not in excess of 400,000 p.s.i. and preferably not in excess of 390,000 p.s.i., with high ductility in excess of 45% in terms of contraction of area before rupture and good properties under elevated temperature conditions in terms of relaxation permanence, the steps of said method comprising, selecting an austenitic stainless steel of the transformation hardening type and containing from 0.01 to 0.20% carbon, up to about 5% silicon, up to about 10% manganese, from 13 to 20% chromium, from 3 to 10% nickel, up to about 25% molybdenum, up to about 25% aluminum and the balance essentially iron; annealing the steel at a temperature of 950°-1100° C. thereby stabilizing the austenite; rapidly quenching the steel from said annealing temperature; cold-working said steel to reduce its cross-sectional area between 40 and 90%, preferably between 60 and 85%; then tempering the steel at a temperature in the range between 200° and 550° C., preferably 250°-450° C. and thereafter cold-working said steel to reduce its cross-sectional area between 5 and 40%.

2. A method according to claim 1, including the step of finally cold-working the steel to reduce its cross-sectional area between 10 and 30%.

3. A method according to claim 1, including the step of tempering the steel at a temperature in the range between 300° and 550° C., preferably between 350° and 500° C., after the final cold working for a period of 2-5 hours.

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UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

PATENT NO. : 3,917,492

DATED : November 4, 1975

INVENTOR(S) : Anders Lars Erik Backman, et al

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In claim 1:

line 10- "0,20%" should read: - 0.20% -

line 12- "25%" should read: - 2.5% -

line 13- "25%" should read: - 2.5% -

Signed and Sealed this  
twenty-fourth Day of February 1976

[SEAL]

Attest:

RUTH C. MASON  
Attesting Officer

C. MARSHALL DANN  
Commissioner of Patents and Trademarks