United States Patent [19]

Hultgren et al.

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[45] **Dec. 31, 1974**

[54] **STEEL PRODUCT HAVING IMPROVED MECHANICAL PROPERTIES**

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[73] Assignee: **Republic Steel Corporation,** Cleveland, Ohio

[22] Filed: **Mar. 19, 1973**

[21] Appl. No.: **342,700**

Related U.S. Application Data

[63] Continuation-in-part of Ser. Nos. 227,045, Feb. 17, 1972, Pat. No. 3,723,144, and Ser. No. 98,674, Dec. 16, 1970, abandoned.

[52] **U.S. Cl.** **148/36,** 148/37

[51] **Int. Cl.** **C21d 9/48,** C22c 39/14

[58] **Field of Search** 148/36, 37, 12

[56] **References Cited**

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Primary Examiner—W. Stallard

Attorney, Agent, or Firm—Cooper, Dunham, Clark, Griffin & Moran

[57] **ABSTRACT**

Steel products, which have been subjected to a desired deformation, are characterized by superior strength, including markedly improved toughness or tensile properties or both, as a result of procedure whereby the steel is made temporarily superplastic at an elevated temperature and is deformed while in such state. This procedure for converting steel, notably ordinary and alloy grades of low carbon, ferritic character, to a superplastic state, e.g. affording very high ductility, and for deforming such superplastic steel in a desired manner, embraces: rapidly heating a body of steel to a temperature, advantageously in the alpha-plus-gamma phase field, where the steel is then found, over a brief interval, to experience a transitional state of severe microstructural instability and to be characterized by superplasticity; and applying stress to the body in such interval to effect the desired deformation. The new products, which are compositionally of the character required for the process, are found to have much higher strength than the original steel.

10 Claims, 17 Drawing Figures

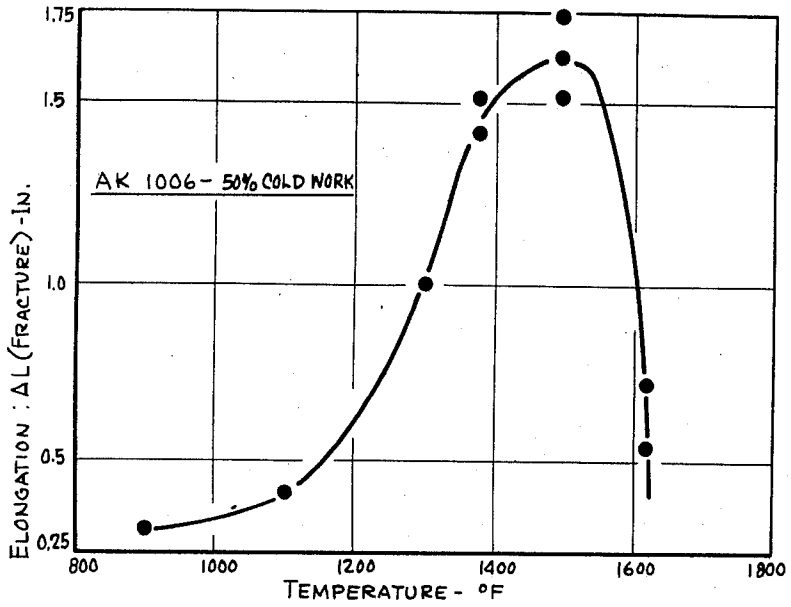
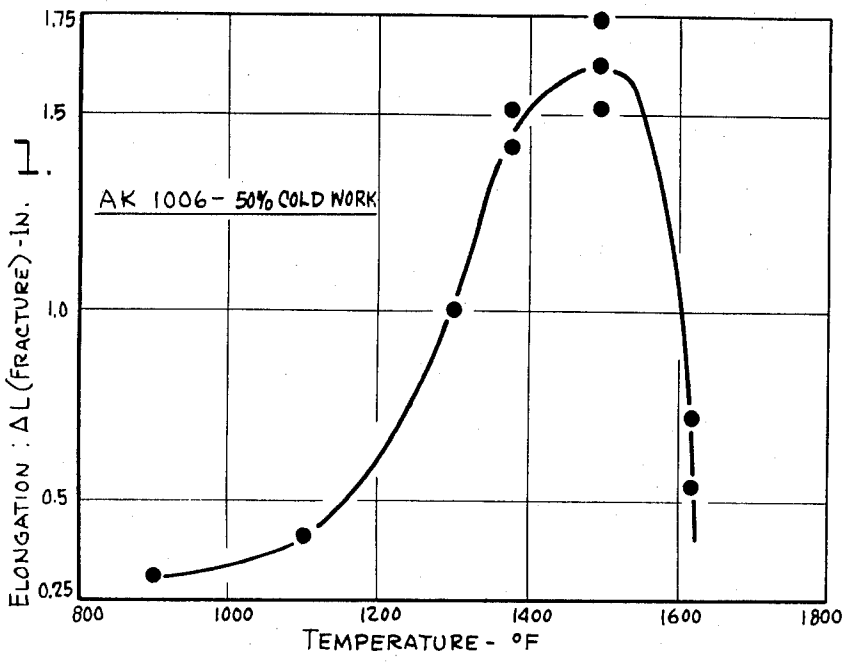


Fig. 1.


1.75
1.5
1.0
0.5
0.25
AK 1006 - 50% COLD WORK
ELONGATION : ΔL (FRACTURE) - IN.
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

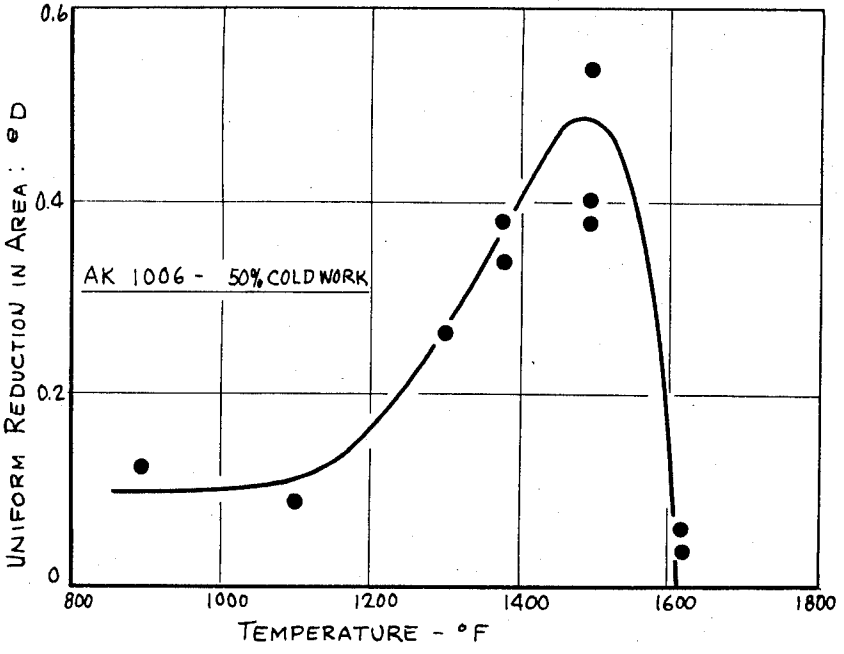
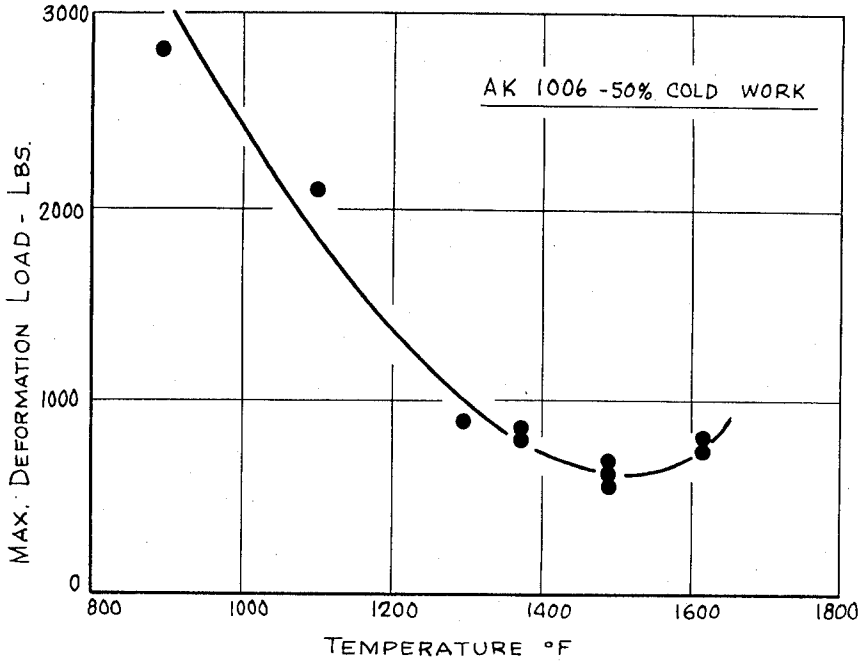

0.6
0.4
0.2
0
AK 1006 - 50% COLD WORK
UNIFORM REDUCTION IN AREA : ε D
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 2.

Fig. 3.


AK 1006 -50% COLD WORK
MAX. DEFORMATION LOAD - LBS.
3000
2000
1000
0
800
1000
1200
1400
1600
1800
TEMPERATURE °F

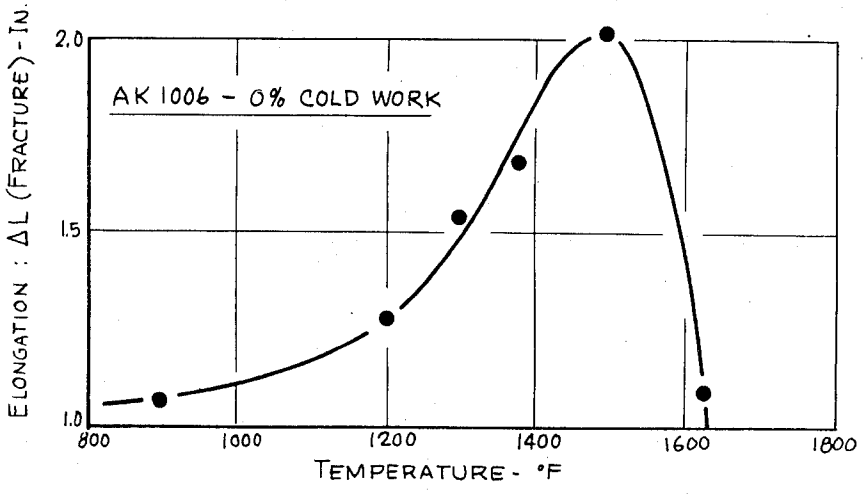

AK 1006 - 0% COLD WORK
ELONGATION : ΔL (FRACTURE) - IN.
2.0
1.5
1.0
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 4.

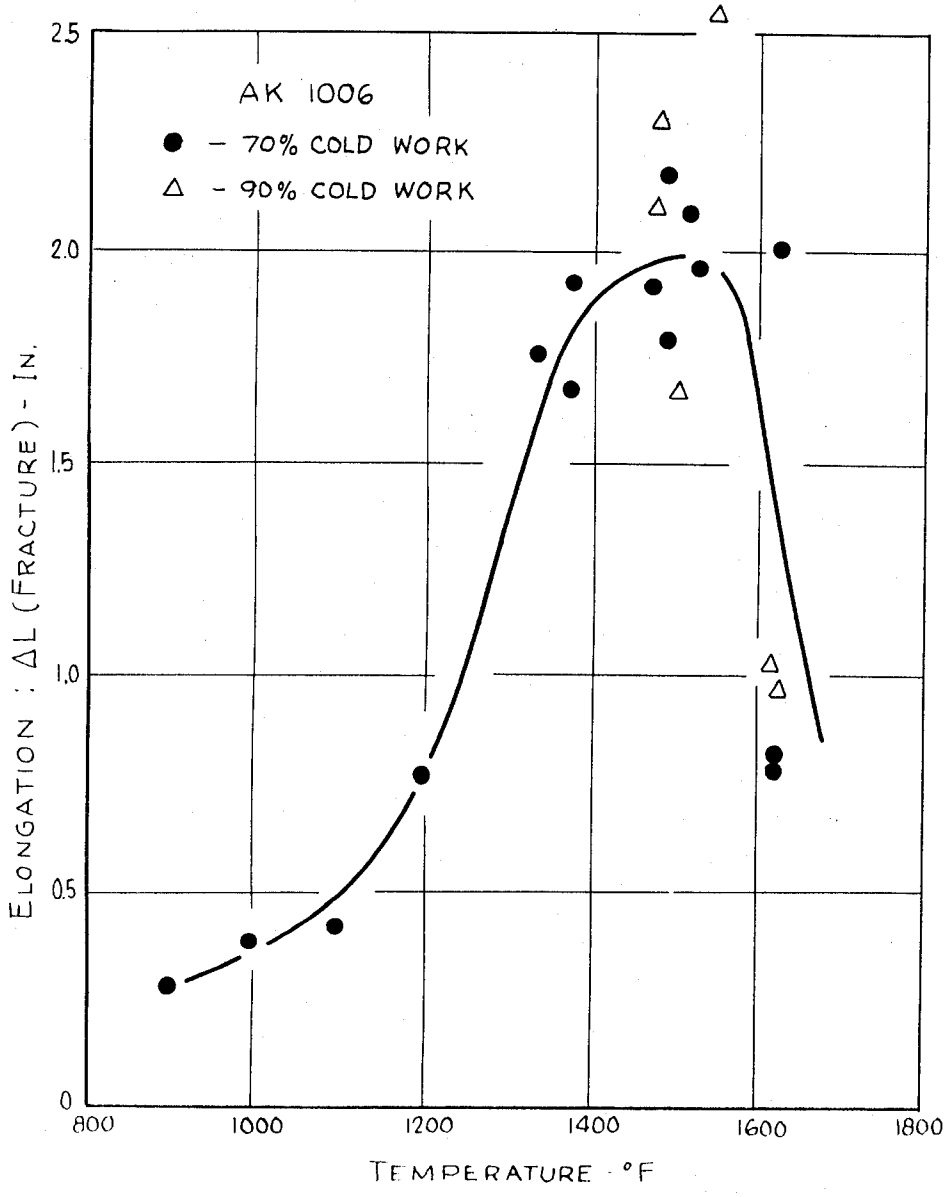

AK 1006
● - 70% COLD WORK
△ - 90% COLD WORK
ELONGATION : ΔL (FRACTURE) - IN.
2.5
2.0
1.5
1.0
0.5
0
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 5.

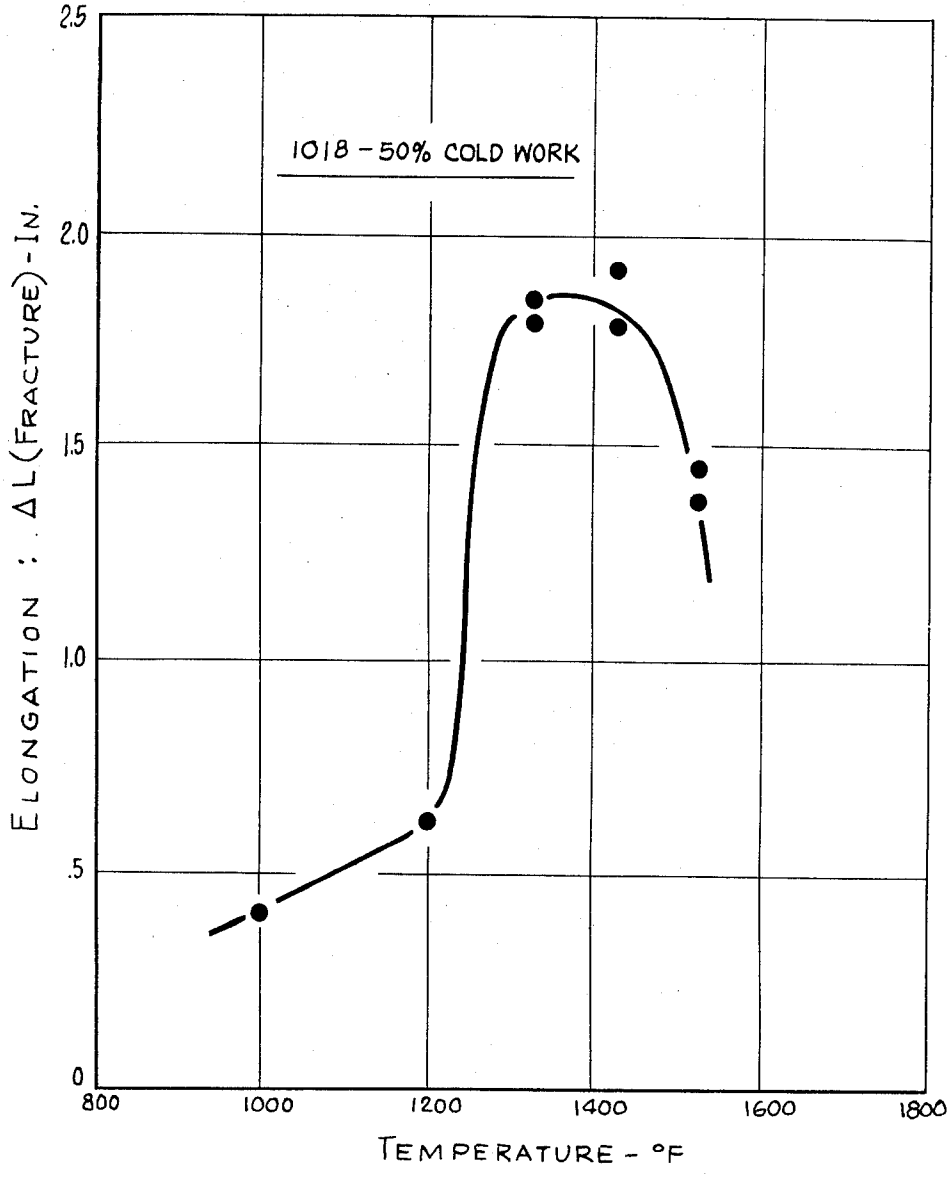

1018 – 50% COLD WORK
ELONGATION : ΔL (FRACTURE) - IN.
2.5
2.0
1.5
1.0
.5
0
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 6.

Fig. 7.

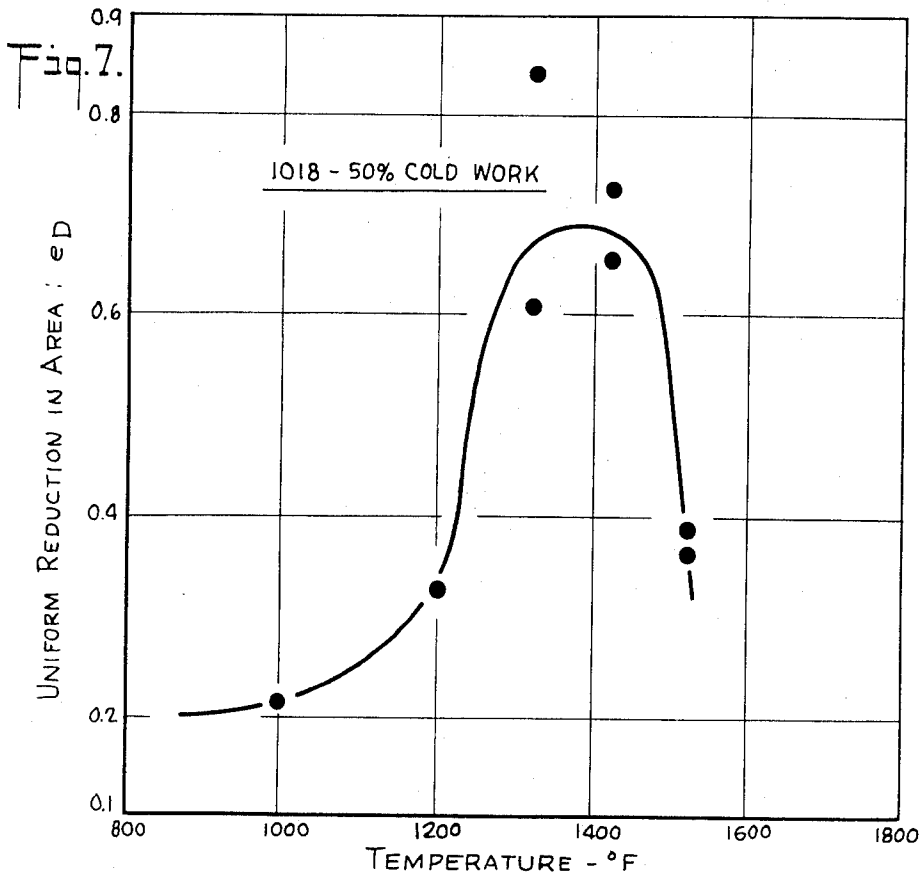

0.9
0.8
0.6
0.4
0.2
0.1
1018 - 50% COLD WORK
UNIFORM REDUCTION IN AREA : eD
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 8.

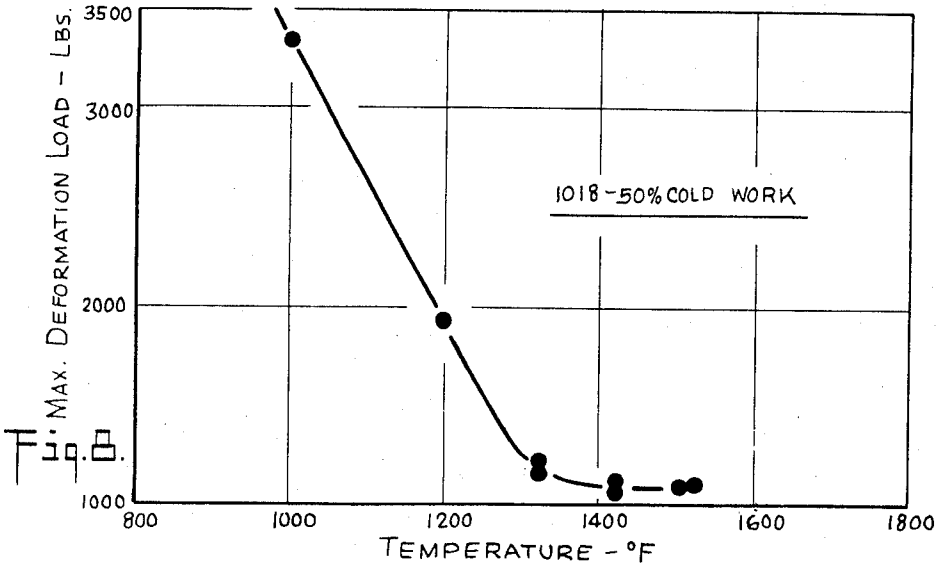

3500
3000
2000
1000
1018 - 50% COLD WORK
MAX. DEFORMATION LOAD - LBS.
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 9.

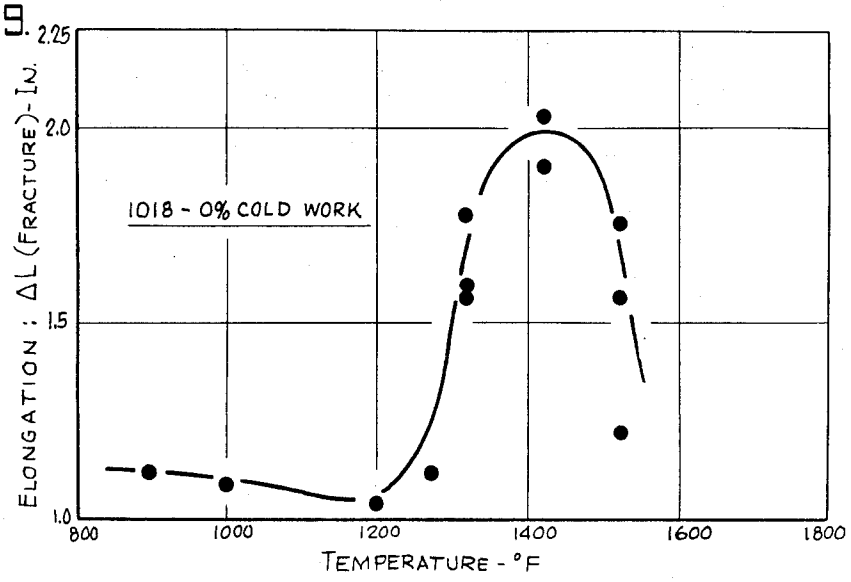

ELONGATION : ΔL (FRACTURE)-IN.
2.25
2.0
1.5
1.0
1018 - 0% COLD WORK
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 10.

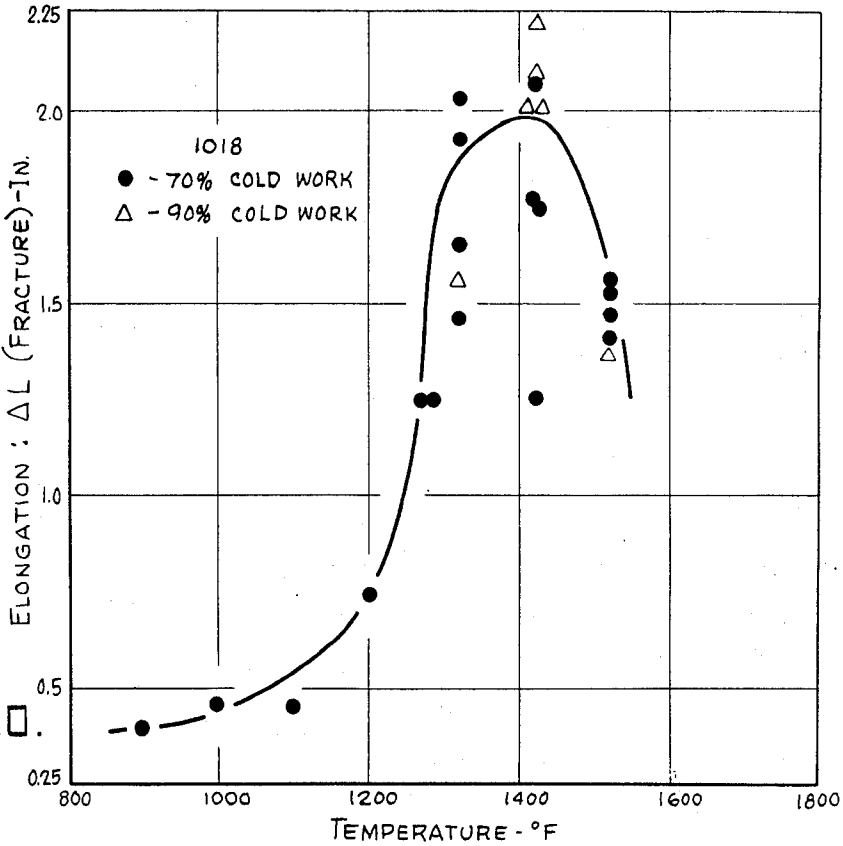
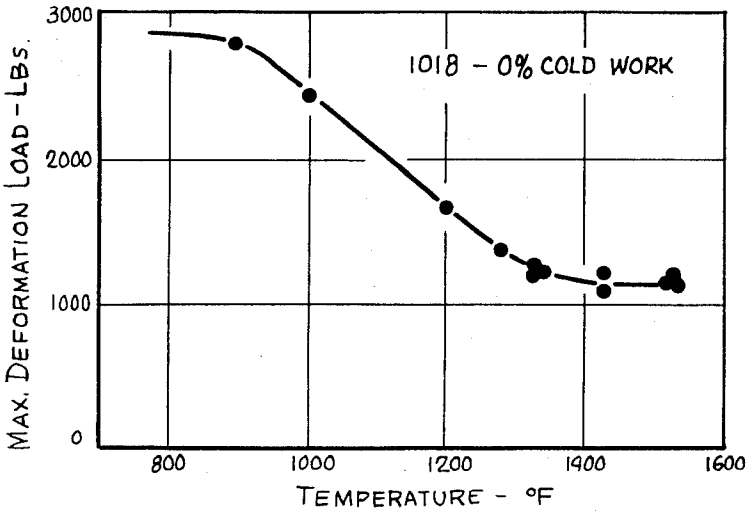

ELONGATION : ΔL (FRACTURE)-IN.
2.25
2.0
1.5
1.0
0.5
0.25
1018
● - 70% COLD WORK
△ - 90% COLD WORK
800
1000
1200
1400
1600
1800
TEMPERATURE - °F

Fig. 11.


3000
2000
1000
0
MAX. DEFORMATION LOAD - LBS.
1018 - 0% COLD WORK
800
1000
1200
1400
1600
TEMPERATURE - °F

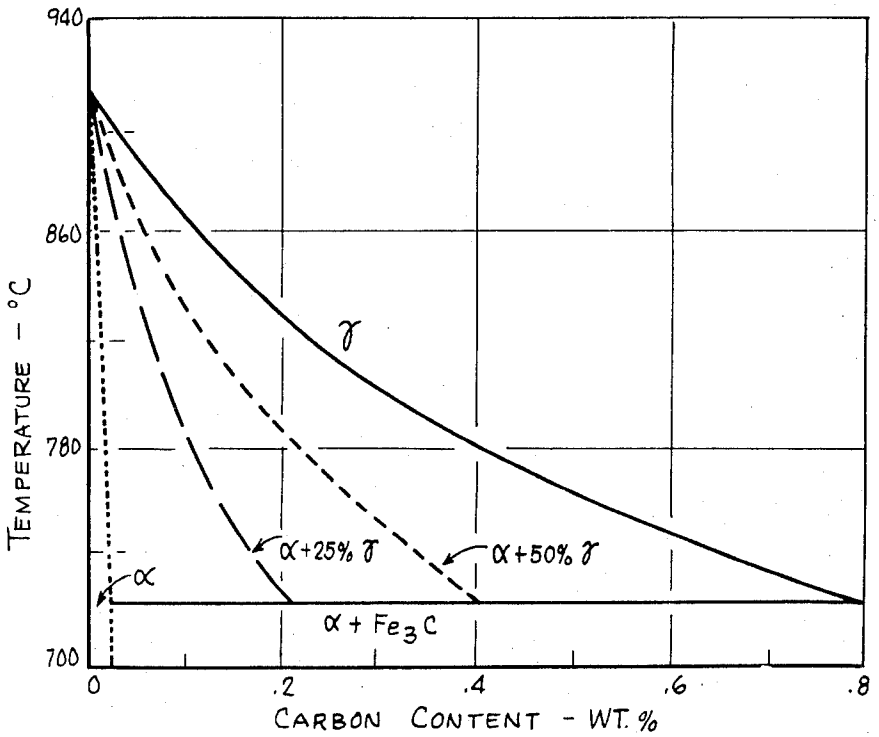

940
860
780
700
TEMPERATURE - °C
γ
α+25% γ
α+50% γ
α
α + Fe3C
0
.2
.4
.6
.8
CARBON CONTENT - WT. %

Fig. 12.

Fig. 13.

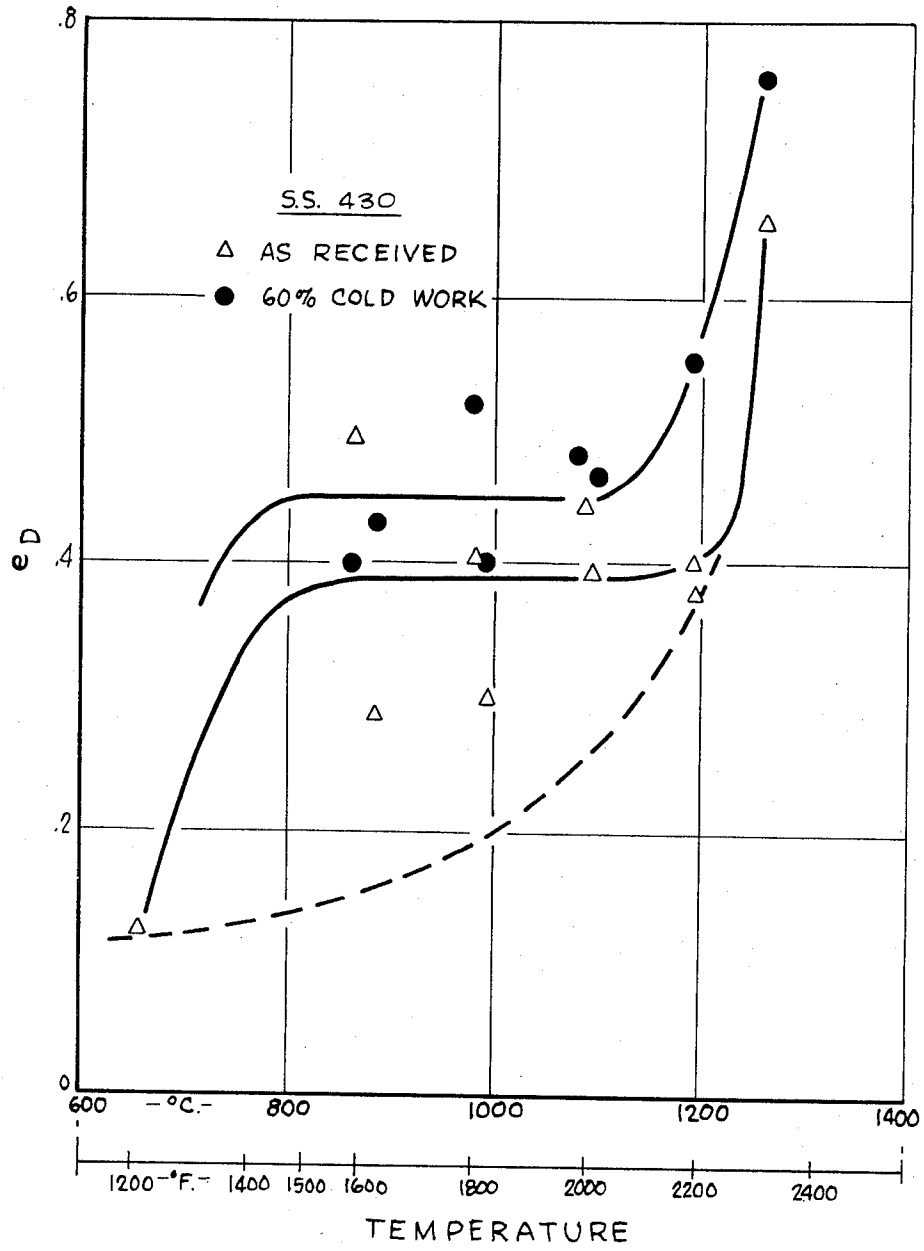

.8
.6
.4
.2
0
ep
S.S. 430
AS RECEIVED
60% COLD WORK
600
800
1000
1200
1400
-°C.-
1200
1400
1500
1600
1800
2000
2200
2400
-°F.-
TEMPERATURE

Fig. 14.

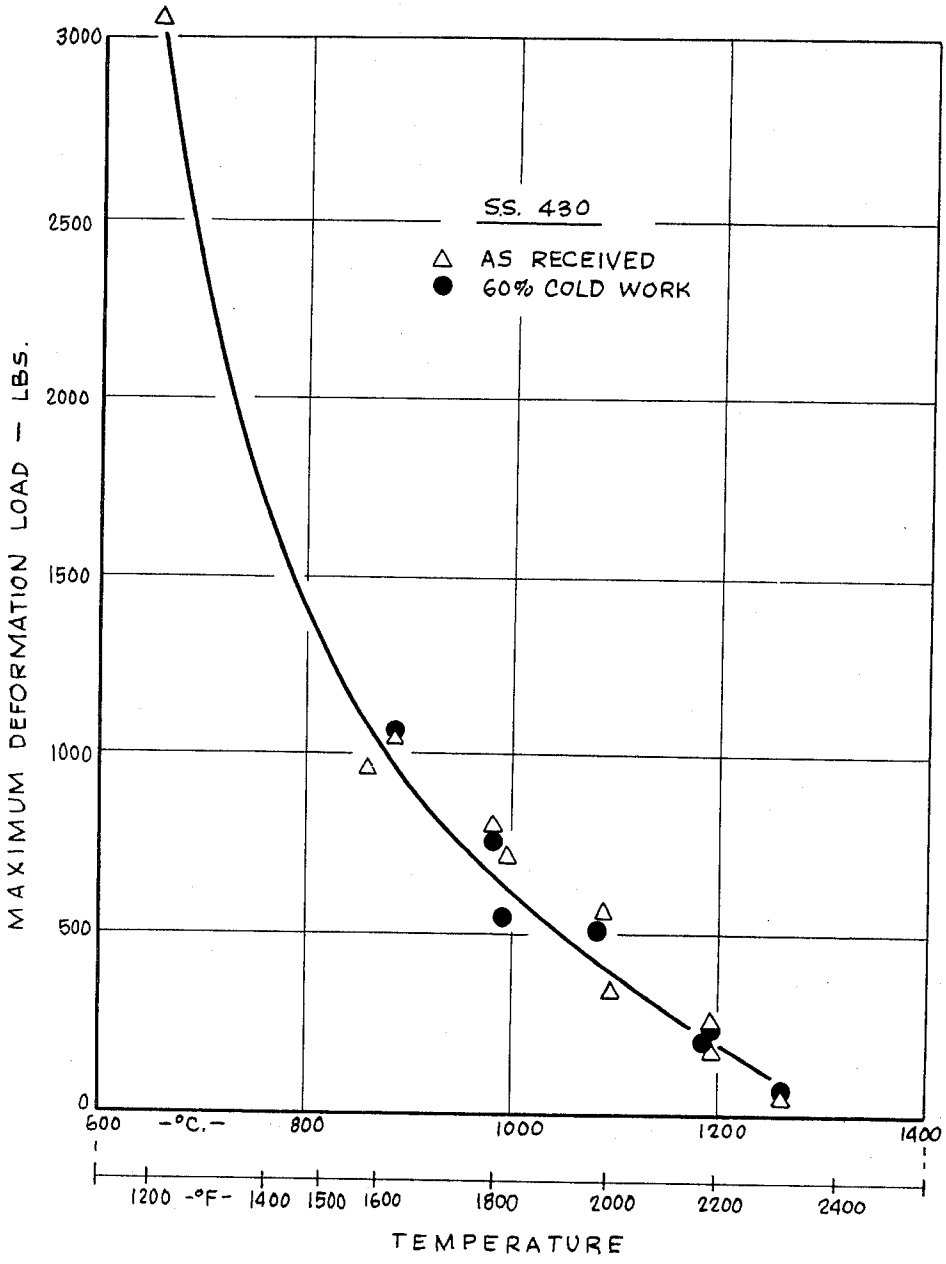
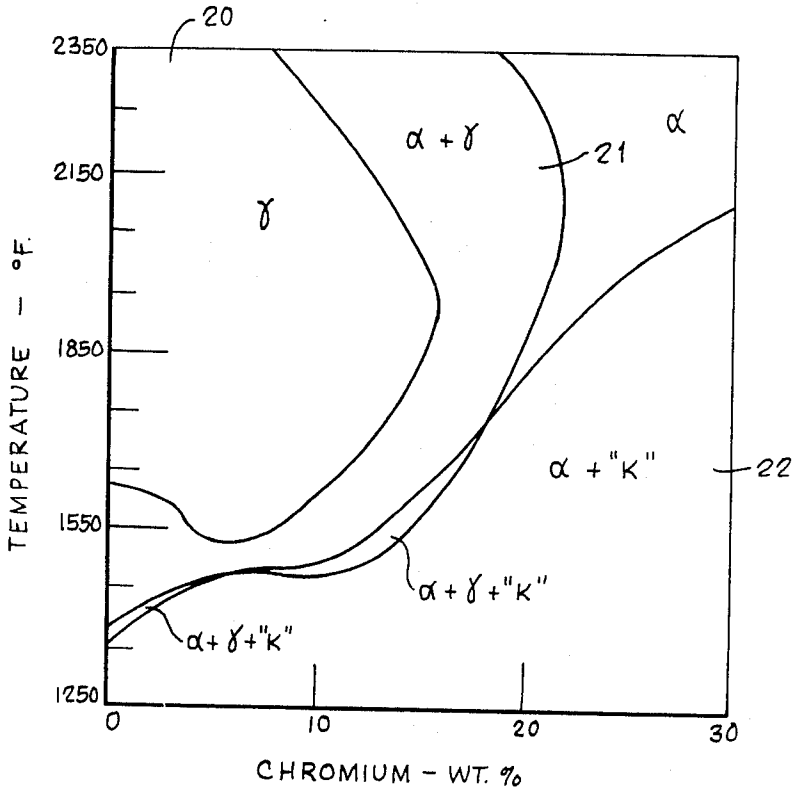

SS. 430
△ AS RECEIVED
● 60% COLD WORK
MAXIMUM DEFORMATION LOAD — LBS.
3000
2500
2000
1500
1000
500
0
600 —°C.— 800 1000 1200 1400
1200 -°F- 1400 1500 1600 1800 2000 2200 2400
TEMPERATURE

Fig. 15.


20
2350
2150
1850
1550
1250
TEMPERATURE - °F.
γ
α + γ
α
21
α + "K"
22
α + γ + "K"
α + γ + "K"
0
10
20
30
CHROMIUM - WT. %

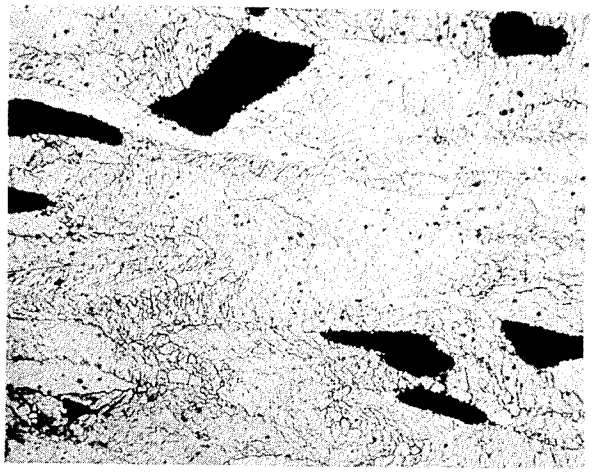


Fig. 16.

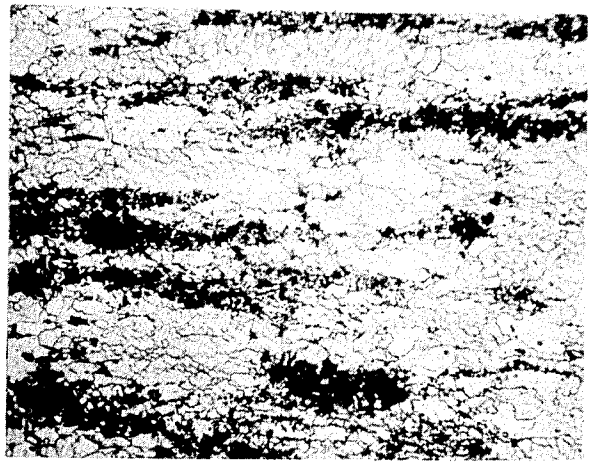


Fig. 17.

STEEL PRODUCT HAVING IMPROVED MECHANICAL PROPERTIES

This patent application is a continuation-in-part of our copending application Ser. No. 227,045, filed Feb. 17, 1972, now U.S. Pat. No. 3,723,194 and of our application Ser. No. 98,674, filed Dec. 16, 1970, now abandoned, with which said application Ser. No. 227,045 was copending.

BACKGROUND OF THE INVENTION

This invention relates to new and stronger steel wherein such as result from new procedure whereby or wherin steel is converted to a superplastic state, e.g. a state of unusually high ductility, and in a more specific and particularly important sense, the invention is concerned with such products derived from the new procedure for making steel and deforming the metal by application of stress to achieve desired characteristics of dimension, shape or the like which may involve a large extent of such deformation. Thus for example elongation by stretching is contemplated up to an extent of the order of **100%** or more and similarly large or difficult changes of shape or dimensions by operations such as pressing, drawing, coining or the like. The improvements are related to a wide variety of low carbon steels including ordinary or so-called standard steels and alloy steels of many conventional compositions, and other normally non-austenitic steels such as a number of stainless steel grades.

Superplasticity is a phenomenon known to be attainable in some metals, that has been used or proposed mostly for certain non-ferrous alloys, such as zinc-aluminum compositions, and aluminum-copper, lead-tin, and tin-bismuth alloys. In general, the techniques used to achieve this state of extraordinarily high ductility, in such alloys, have been directed to establishing an extremely fine grain or microstructure, as by recrystallization with or without precipitation effects; a stable condition of superplasticity is then attained for the desired large extent of deformation. Another principle sought to be utilized is to take some advantage of change of phase in metals characterized by different phase conditions, one expedient being to apply heat and cooling so that the metal object is cycled slowly back and forth through a phase change region while stress is applied over a relatively long time, to effectuate deformation.

Although it is readily seen that the attainment of superplasticity or extreme ductility in steel would permit an expedited and simplified mode of fabricating a variety of articles where large changes of dimension or shape are required from an initial piece or blank, or where an article could be made in one piece to have a complex configuration of relatively thin section, not heretofore so produced, there has been no practical success, as presently known, in achieving superplasticity with steels of ordinary types, particularly those mentioned above.

Whether attempted by utilization of the techniques described for non-ferrous metals or by some similar or other approach, superplasticity of practical and commercially useful nature does not appear to have been reached in any ordinary steels, especially those that at room temperatures or other normal temperatures of use are essentially non-austenitic. A principal difficulty is that when superplastic steel bodies have been established, capable in theory of very large elongation under

stress, the strain rate attainable for such elongation (i.e. without rupture) has been extremely low, e.g. of the order of 0.0001 to 0.1 per minute. It will be understood that strain rate is a measure of the rate or speed of elongation, determined as the ratio (e.g. in inches per inch) of the increment of extension to the original unextended length, per unit of time. Strain rates as low as 0.1 per minute, and notably the still lower rates mentioned above are such as to have essentially no practical or commercial value. Formation of a single article to have anything like a deformation of the order of 100% would require a time interval of at least many minutes, and most likely of the order of an hour or hours, which is manifestly not feasible.

It will be understood that references to deformation and elongation as related to desired superplasticity or ductility, are herein concerned with substantially uniform changes of length or section over the entire extent of the article subject to deformation. This is in contrast to the ordinary situation of steel where, for example, an application of tension, as in standard tensile testing of a bar or rod, results first in moderate degree of uniform stretch, and then produces a phenomenon known as necking, whereby a localized reduction of sectional area occurs, producing a very thin region at a single place, further stress then leading to rupture at such locality. The present procedure is related to superplasticity in the sense that deformation or reduction of cross-sectional area occurs substantially uniformly throughout the length of a piece being stretched, without necking or other appreciable local consequences — i.e. short of ultimate rupture at the ultimate limit of superplastic elongation.

The invention is therefore designed to afford what are believed to be novel characteristics in steels, particularly to achieve a significant condition of very high ductility, and further and most particularly, to provide steel of new properties from these novel forming or shaping operations, producing dimensional changes or other changes of configurations, in a single step, not heretofore generally possible. As will be understood, a primary feature of the invention is to attain the stated superplasticity with the characteristic of a practical strain rate, e.g. upwards of one per minute and indeed desirably in the range of 100 to 1,000 per minute.

SUMMARY OF THE INVENTION

The present invention relates to the products of the process herein described, having unusual advantage in respect to strength, notably toughness as measured by Charpy V-Notch impact values. According to the process, the steel is deformed upon establishing superplasticity in it by a relatively simple treatment which develops a high order of microstructural instability in the metal, and permits rapid application of deforming stress in substantial coincidence with such instability.

An essential feature of the process involves heating the steel, as from room or other ordinary low temperature, extremely rapidly to an elevated temperature, specifically a temperature where the alpha-to-gamma transformation can then proceed while the steel is thereafter held at such temperature for a short but convenient interval. In particular, the temperature is advantageously selected as one which would afford, at equilibrium, a combination of the alpha and gamma phases of iron, e.g. such as characterizes the well-known alpha plus gamma phase in the conventional

temperature-versus-carbon equilibrium phase diagram for ordinary steel. That is to say, it has been found that instead of attempting to achieve a stable or temporarily stable condition of very fine grain size or the like, or instead of endeavoring to cycle the metal somehow through variations in equilibrium condition between one phase and another, it has been found that the attainment of a severely unstable microstructure — specifically by the procedure of rapid heating upward to a selected elevated value and then holding at said value for a short interval while relatively rapid deformation is effected — an unexpectedly very high ductility is attained. When formed, moreover, the metal of the article promptly proceeds to an equilibrium or stable condition, and the resulting piece, e.g. upon cooling in any desired manner, reattains substantially the characteristics of the selected steel composition with its normal mechanical and other desired properties, or even in some instances superior properties. There is thus no need for any special step or operation to terminate or destroy the superplastic condition, after the forming step.

In many instances, the process of the invention can be circumstanced or modified to take advantage of two cooperating mechanisms or phenomena that coact to establish a special degree or extent of superplasticity. Thus by first subjecting the steel to drastic cold reduction, as for example cold rolling to an extent of 70% or preferably higher, e.g. 90% reduction, the process is capable, indeed inherently by virtue of the range of temperatures selectable for the above-described unstable state, of providing a correlated characteristic of instability, with respect to recrystallization and with phase-change instability.

This special procedure may be explained by reference to examples, as in the use of ordinary low carbon steel, e.g. AISI 1006 or AISI 1018, respectively 0.06% and 0.20% carbon. The steel, after the usual first stages of production and hot rolling, is cold rolled to 90% reduction, and then the desired piece, without annealing, is rapidly heated, as at a rate of 100° to 200° F. per second, to a temperature within the alpha plus gamma phase area, advantageously midway in such area, this being the area where the ferrite phase begins substantial transformation to the austenite phase and indeed where at equilibrium, e.g. over a period of time, the structure would consist of both ferrite and austenite. In accordance with the procedure of the invention, the article is held at the selected temperature for a short interval, say five seconds, and then subjected to rapid deformation, as by drawing, stretching or pressing, to the desired large extent. Conveniently the total time of continuing deformation may be relatively short, of the order of a few seconds or less.

In this way a high degree of microstructural instability occurs in the piece as it is held at the selected temperature, notably after the stated holding interval, and the instability is characterized, according to present understanding, by the initiation and continuance of extensive recrystallization simultaneously with the rapid, vigorous, phase transformation from ferrite toward and to austenite. During and as a heretofore unappreciated result of these conditions of severe instability, a substantial state of superplasticity, i.e. a condition of very high ductility, is found to occur and thus to permit the desired deformation.

As stated, the invention is nevertheless basically advantageous, according to present understanding, without the cold reduction of the metal and thus can be applied to steel produced without cold reduction or to steel which has been cold reduced and annealed, even though in such cases reliance for superplasticity is then placed primarily on the high degree of transformation instability after rapid heating to the selected temperature. It is presently believed, moreover, that in at least some instances the superplastic property may be enhanced by what can be called a dynamic recovery brought about by the fact of elongation or other deformation — i.e. a secondary (second occurring) phenomenon or mechanism which is advantageously occurring in all cases.

As used with any of a large variety of steels, most advantageously low carbon steels but also alloy steels and likewise non-austenitic stainless steels, e.g. the compositions classed as ferritic and martensitic in the 400 series, the procedure can be effective for the production of many highly or intricately deformed shapes, with great economy and efficiency.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGS. **1** and **5** inclusive are graphs where various significant values of deformation properties are plotted against testing temperature, being temperatures to which specimens of a selected steel, AISI 1006, were very rapidly heated and then held, in accordance with the invention, these graphs showing the unusual results attained at the temperature of severe microstructural instability, as follows:

FIG. **1** being a plot of elongation to fracture, against testing temperature, for the steel which had been cold worked to reduction of 50%;

FIG. **2** being a plot of uniform reduction in area (i.e. non-necking reduction) against testing temperature, again after 50% cold work;

FIG. **3** being a plot of maximum deformation load against testing temperature (50% cold work);

FIG. **4** being a plot of elongation to fracture, like FIG. **1**, but for specimens which had not been cold worked; and

FIG. **5** being a plot of elongation to fracture, like FIG. **1**, for groups of specimens that had respectively been cold worked 70 and 90%.

FIGS. **6** to **11** inclusive are graphs of deformation properties against testing temperature, similar to FIGS. **1** to **5**, for specimens of another steel, AISI 1018, as follows:

FIG. **6** being a plot of elongation to fracture, for specimens having 50% cold work;

FIG. **7** being a plot of uniform reduction in area, 50% cold work;

FIG. **8** being a plot of maximum deformation load, 50% cold work;

FIG. **9** being a polot of elongation to fracture, for specimens with no cold work;

FIG. **10** being a plot of elongation to fracture, for groups of specimens that had respectively been cold worked 70 and 90%; and

FIG. **11** being a plot of maximum deformation load, for specimens with no cold work;

FIG. **12** is an iron-carbon equilibrium phase diagram, plotting temperature against carbon content for pure iron-carbon systems, showing selection of temperatures especially suited for practice of the invention, e.g. for

the hypoeutectoid compositions there are two intermediate boundaries shown which predict mixtures of 75% alpha, 25% gamma and 50% alpha with 50% gamma, each so labeled in the diagram;

FIG. **13,** illustrating deformation properties in accordance with the invention, is a plot of uniform reduction in area against testing temperature for specimens of stainless steel grade .430, both in hot rolled condition and after 60% cold work;

FIG. **14** is a plot of maximum deformation load against testing temperature for specimens of stainless steel grade 430, as in FIG. **13.**

FIG. **15** is an equilibrium phase diagram for stainless steels of the 400 series, plotting temperature against weight percent chromium, exemplified at a carbon level of 0.05%.

DETAILED DESCRIPTION

More particularly explained, the procedure is notably applicable to so-called hypoeutecoid compositions, meaning compositions where the carbon content is less than the eutectoid value, e.g. 0.8% in ordinary low carbon steel. Preference for this low carbon range is related to the fact that in the ordinary equilibrium diagram plotting carbon percentage and temperature for such steels, FIG. **12,** there is an area (commonly designated by the letters alpha plus gamma, and here bounded by the gamma, alpha and alphacementite regions) in a temperature range from generally about 1,350° F. to varying higher points up to about 1,650° F. (about 725° to 900° C.), where a stable structure can be reached having a composition consisting of both alpha and gamma phases of iron. It will be understood, however, that the procedure of the invention in no sense involves the attainment of such equilibrium for providing superplasticity, but on the contrary resides in the discovery that a severe microstructural instability, which in turn is found to exhibit the desired superplasticity, occurs when the steel has been heated rapidly from low temperature (meaning any temperature sufficiently below the selected value as not to involve transformation and as not to involve recrystallization where there has been substantial cold reduction) up to the selected value and then holding at said value for development of such instability and for employment of it, so to speak, in deformation under stress. A particularly important consideration is that in this alpha-plus-gamma zone, the instability is severe, and though brief, lasts long enough to accommodate the desired, rapid operation of deformation.

The new steel products, e.g. characterized by greatly increased strength as compared with the untreated steel, have compositions as described herein for suitability of application of the process, including the hypoeutectoid steels. The products have much improved impact strength, or better yield and tensile strength, or enhanced properties in both respects, and reveal a distinctive microstructure. Further details of the procedure of treatment and deformation are here given, followed by data about the special properties of the resulting products.

Thus the invention is specially applicable to steels, such as the above mentioned hypoeutectoid low carbon standard steels, where the equilibrium diagram exhibits and area of coexistence of two phases, e.g. both ferrite and austenite, or as more generally represented on such phase diagram, an area representing, at equilibrium, a combination of alpha iron and gamma iron. Whereas in ordinary steels the eutectoid is a composition containing about 0.8% carbon, corresponding characteristics are shifted or different in alloy steels and notably in the non-austenitic stainless steels, e.g. so-called ferritic or martensitic steels of the 400 series. Nevertheless, it is apparent that an austenite-ferrite area exists in a wide variety of steels, notably of low carbon content, and can be availed of for optimum practice of the invention, by corresponding selection of a terminal and holding temperarture after the stated fast heat-up, for achieving a high order to temporary instability.

To a degree, however, it is also conceived that the procedure can be utilized with other steels, advantageously of low carbon content (e.g. not more than 1%), where a value of elevated temperature can be selected and maintained such as to yield a state of transformation instability which, even though very short, may be sufficient to permit deformation under stress. As will be understood, however, selection and maintenance of such temperature may require special care, notably to avoid so rapid a phase transformation as not to permit practical utilization of the instability of microstructure in accordance with the principles explained above. Moreover, if advantage is also to be taken of the action of recrystallization while it is going on — such action being conceived as the conversion of large, elongated crystal structure resulting from cold reduction, to a fine, equi-axed crystal form — care must be taken to time the occurrence of such action with the occurrence of the transformation instability.

The procedure can afford an unusual forming operation effective for production of a variety of articles such as greatly elongated parts, cuplike elements, or similar tubular casing or pieces closed at one end having cylindrical or other shapes, various other hollow articles such as refrigerator shells, hub caps, automobile body elements, and the like. In general, these are parts which may have heretofore required a series of forming steps, sometimes a long series, as by successive stages of cold forming, e.g. pressing, drawing or other deformation, interspersed with annealing steps. Inasmuch as the strain rate afforded by the invention generally resides in the range of 100 to 1,000 per minute and inasmuch as the property of superplasticity is found to be accompanied by a marked decrease of the required deformation load, the process results in an unusually rapid and highly economical operation for fabricating finished or semi-finished structures in one or no more than a few forming steps, from a wide variety of steels of recognized commercial type, including steels of essentially inexpensive character such as the ordinary low carbon compositions.

Referring further to specific examples of the invention, an extensive series of tests was carried out with specimens of ordinary carbon steels as noted above, being AISI 1006 (0.60% carbon) and AISI 1018 (0.20% carbon), both well below the eutectoid. The 1006 steel was aluminum killed, but presence or absence of such condition is not understood to bear significantly upon the invention. The steels had the usual manganese contents (below 0.39% and 0.67% respectively) and were entirely conventional nonalloyed compositions, with usual limits of phosphorus and sulphur. Ordinary processing with hot rolling had been followed in each case.

For test purposes, cylindrical specimens were prepared, ¼ inch diameter by 8 inches long, by machining from suitable pieces. Where cold working was employed prior to test, the pieces were of material which had been reduced by cold rolling in conventional manner, without intermediate or final anneal. SPecifically, specimens were employed for which the metal had zero cold working and cold working to reductions of 50, 70 and 90% respectively. These percentages refer to reduction in thickness, the cold rolling being effected with the necessary series of passes, preferably in the same direction, for production ease. In order to take full advantage of the occurrence of recrystallization in practice of the invention, the selected degree of cold working should be performed without intermediate or subsequent anneal; any previous treatment (not employed in these tests) which may have involved a cold work and anneal, is disregarded. For the invention, cold working can be achieved in any suitable manner, as by rolling, forging or like operations.

All of the tests in these series involved heating the specimen rapidly, i.e. at a rate of 120° F. per second or thereabout, to a test temperature, where the specimen was held for a predetermined short time, which was 5 seconds for the 1006 specimens and 10 seconds for the 1018 specimens, and then each specimen (while remaining at such temperature) was subjected to rapid deformation, e.g. at a cross head speed of 90 inches per minute. The equipment used, for heating, holding and deformation, was an apparatus commercially available for test purposes (under the name of Gleeble), designed for other tests of high temperature properties as in welded structures.

In general, the tests involved determination of deformation characteristics at various test temperatures, being the selected temperature to which the specimen was heated, and at which it was held, and the results demonstrated the occurrence and utilization of superplasticity in accordance with the invention, being the effect of severe microstructural instability when the temperature was in the austenite-ferrite range for these steels. For best representation of superplasticity, all deformation tests in these examples were of tensile characteristics, and all deformation was effected by application of tensile stress alone, exerted between elements gripping the end portions of the specimens. It was fully apparent, indeed confirmed by other investigation, that the extraordinarily high ductility revealed by these tests was accomplished by other, unusually superior properties of deformability, as in pressing to deep or complex shapes, full conformity with intricate die configuration, and the like.

One index of superplasticity was taken to be the extent of elongation to fracture, under tensile stress. For simplicity of comparison among specimens of identical shape and size, identically held by the tension-applying elements of the test apparatus, this elongation was simply measured as the total increment of length, in inches, exhibited by the stretched specimen, to the point of fracture. The fact that in each case the specimen necked down, just prior to fracture, at the fracture locality did not affect the comparative significance of the results, it being fully apparent that the large values of increment in length obtained at the test temperatures of the invention were fairly representative of a large comparative increase in neck-free elongation of the free central portion of the test specimen which had an effective gauge length of about one inch.

FIG. 1 shows the result of such tests for various specimens of the 1006 steel that had been cold worked 50%, the specimens which were prepared from the cold worked metal being rapidly heated to various test temperatures as shown in the figure, held and subjected to stress deformation as described above. As will be seen, a very pronounced maximum of elongation to fracture was achieved for temperatures in the range of about 1,375° to about 1,550° F., with optimum results at approximately 1,500° F. These values coincide with the alpha-plus-gamma region of the equilibrium diagram (see FIG. 12), the value of 1,500° F. (about 815° C.) being substantially that for which at equilibrium, e.g. after prolonged holding and stabilization the metal (0.06% carbon) would consist of approximately 75% ferrite and 25% austenite from the figure for pure iron-carbon. The actual phase boundaries for commercial alloys will be shifted due to the presence of Mn, P, S, Al, Si, etc.

Another and somewhat more significant value was measured as indicated in FIG. 2, being the extent of uniform reduction in area, obtainable under tensile elongation, at the several test temperatures. Specifically this is in effect a measure of the maximum cross-section reduction which is reached in each case before necking occurs. This is believed to be an effective measure of superplasticity, or of high ductility, inasmuch as the purpose of the invention is to achieve a high order of deformation, without necking or equivalent localized effect in the workpiece subjected to the desired deformation. The value actually determined in these tests is a function of the ratio of the original cross-sectional area of the test rod to the final area (just prior to necking), conveniently a logarithmic value e_D, defined as

$$e_D = 2\ 1n\ (D_o/D_f)$$

where D_o is the initial diameter and D_f is the final diameter, with factor 2 providing the conversion to a ratio of areas. The values of e_D obtained at various test temperatures are shown in FIG. 2, where the test specimens (of 1006 steel) were the same as in FIG. 1. Percent elongation is definable in reference to e_D data; thus e_D values of 0.4 to 0.8 represent uniform elongation of about 50 to 125%. Normal uniform elongation for these steels is about 25–30% or less.

It will be noted that in FIG. 2 a sharp maximum of useful result again appeared at about 1,500° F., with useful high values also apparent at adjacent points within the range of the austenite-ferrite region. Specifically the measured quantity e_D peaked to values varying from 0.4 to above 0.5 (i.e. above 65% elongation) at the effective temperature range from values of 0.1 (10% elongation) and less both below and above it.

A further and extremely desirable characteristic of superplasticity is a large reduction in required deformation load, i.e. stress required to deform the workpiece. With the specimens of the 1006 steel which had received 50% cold work, measurements were made of the maximum load needed for tensile deformation at various test temperatures, i.e. the maximum reading of applied load reached during the brief interval of deformation. Results were as plotted in FIG. 3, showing a low minimum of such deformation load at about 1,500° F., with a useful range of superplasticity generally similar to that exhibited in the preceding figures. Specifically,

the required tensile load dropped to well below 1,000 pounds, indeed close to 500 pounds, from values in the range of 2,000 to 3,000 pounds at lower temperatures. Other investigation has indicated that the maximum tensile load likewise rises at higher temperatures than as shown in this graph; certain limitations of the test equipment prevented extension of this and other curves about 1,600° F. in these sets of tests.

Tests of elongation to fracture, similarly made and measured as those reported in FIG. **1** and following identical procedure of treatment of the specimens were also made of specimens of the 1006 steel where the prior treatment of the metal was different from that in FIG. **1.** Specifically, tests on specimens of steel that had received no cold working are reported in FIG. **4,** showing a significant maximum of ductility at essentially the same value, i.e. about 1,500° F. Like tests on specimens of which the steel had received relatively large amounts of cold working (without anneal) are plotted in FIG. **5,** the solid circles representing specimens of steel had been cold reduced 70%, and the triangles being specimens of metal that had received cold reduction to 90%. The superiority in elongation to fracture, at and adjacent to the optimum temperature of 1,500° (reaching increments well above 2 inches) is very great, being especially so for the 90% cold worked metal.

The second example of the invention is illustrated by the tests similarly made with specimens of the 1018 steel as explained above. For metal that had been cold worked to 50% reduction, FIG. **6** shows the values of elongation to fracture (maximum nearly 2 inches), while FIG. **7** correspondingly shows the extent of uniform reduction in area (maximum e_D, 0.6 to 0.8+), and FIG. **8** indicated the maximum deformation load (lowest 1,000 pounds), all for various test temperatures. The curves and results agree qualitatively with those in FIGS. **1, 2** and **3,** indicating a very large increase in ductility and a large decrease in required tensile stress, at the optimum temperatures for this alloy, specifically a temperature in the range of about 1,350° to 1,450° F., notably temperatures around 1,400° F.

Again, tests of the 1018 steel which had not been cold worked at all and which had received higher degrees of cold working were made, under conditions otherwise the same as those for FIGS. **6.** Specifically, in FIG. **9,** the steel without cold working showed a fairly sharp maximum of elongation to fracture at around 1,400° F. (peak value about 2 inches), while very high values were achieved in the steels tested in FIG. **10,** the solid circles being 70% cold worked material and the triangles 90% cold worked material (peak values well over 2 inches), the optimum temperatures, around 1,400° F., being clearly similar to those revealed in FIGS. **6** to **9.**

Corresponding to FIG. **8,** tests of maximum deformation load (for tensile stress) were made as reported in FIG. **11,** relative to the 1018 specimens (see FIG. **9**) of which the metal had received zero cold working. A minimum value (about 1,000 pounds, as against other values up to 3,000 pounds), being an extremely desirable attribute of superplasticity, appears in the vicinity of the optimum temperature of 1,400° F.

From all of these tests, which in effect represent practice of the invention at the optimum and near-optimum temperatures, and from other investigations by test, it has been demonstrated that the present procedure affords unusually high ductility, representative of the desired superplastic state, and thus permits feasible and rapid deformation to large uniform elongations, such as 50% and well above, and usually of the order of 100% and greater, in optimum conditions. Effective results are achieved with material which has received no cold working at all, but significantly superior advantage is demonstrated for metal cold worked to a reduction of about 70% or more. It will be seen that the process is eminently practical, requiring only a rapid heating of the piece up to the selected deformation temperature, then holding the piece at such temperature for a brief time to develop severe microstructural instability, and rapidly performing the desired deformation, indeed under relatively low stress.

Summarizing the above examples and taking into account other experimental work of similar sort relative to the invention, a presently preferred practice, for obtaining excellent deformability at high strain rates in the case of ordinary carbon steels having a carbon content below 0.8% is as follows: the piece is heated rapidly at a rate of 100° to 200° F. per second to a selected temperature in the two-phase field, preferably a temperature which on holding to the point of stabilization would produce a microstructure of about 50% ferrite and 50% austenite. Thereupon after a short holding time, e.g. 5 to 10 seconds, the piece is deformed under stress, conveniently so at a strain rate upwards of 10 per minute and advantageously in the range of 100 to 1,000 per minute. Maximum superplasticity appears to be obtained if the steel has previously recieved a significant cold reduction, e.g. 70% and upwards, and preferably about 90%. For the AISI 1006 steel, the selected temperature (apparent optimum 1,500°) can be in the range of 1,475° to 1,550° F., while a preferred value for AISI 1018 is about 1,400° to 1,425° F. It may be noted in reference to the above tests as involving 90% cold reduction, this aim was not in fact precisely reached, i.e., reduction was only about 85%, but it is clear that the results can properly be stated as those characterizing the effect of approximately 90% reduction.

It is essential that the treatment temperature be reached by heating the metal up from a much lower value, conveniently from room temperature or usually at least from a value several hundred degrees F. below the transformation point. Indeed where recrystallization effects are to be significant, the heat-up must progress from a temperature well below any annealing value. The rate of heating is preferably quite rapid, as in the range mentioned above, although it is understood that substantially lower rates can be useful, especially where the onset of severe instability does not occur before reaching the desired temperature. In general heat-up rates should be at least 10° F. per second, more suitably 20° F. per second and upwards; a good range appears to be from 50° to 300° F. per second. While there is no theoretical upper limit, convenience is served by rates below 500° per second; and indeed in some cases a lower degree of superplasticity has inexplicably been noted with extremely fast heat-up times, e.g. above 1,000° F. per second. The time taken, from room temperature, has normally been of the order of 10 to 12 seconds, with little change in result at times as high as 60 seconds or as short as 1 or 2 seconds. Correlation of the heat-up rate with occurrence of recrystallization (if desired) from cold worked condition is relatively simple to determine, usually in the light of known

or ascertainable characteristics for annealing the selected steel.

In most cases, some holding time will be required, suitable values for the low carbon steels having been indicated above. In other situations the optimum time is at most a matter of simple test. For example, after selecting a conveniently rapid heat-up time and selecting an appropriate terminal temperature from the phase diagram for the metal (where the minimum is usually about 1,330° F. for ordinary steel) a short series of tests, e.g. utilizing several different holding times and then applying deformation at the desired strain rate, will readily reveal the holding interval for best results. Indeed selection of the treatment temperature itself may be correlated with the strain rate needed for a given type of deformation or other needs, in that within an available range of temperature, the useable range of strain rates appears to rise with temperature.

Once the parameters have been selected, as to treatment temperature, heat-up rate and holding time, practice of the invention is a convenient and expedited operation. Suitable provision is made for heating the workpiece rapidly, as by induction heating, and then by appropriate thermal control, maintaining the piece at selected temperature for the desired time, including the holding interval and the very brief period required for actual deformation. The latter task may be performed in any suitable fashion, with equipment adapted to hold the piece at the desired temperature, e.g. as part of the heating equipment, or to receive the piece from such equipment into whatever dies (e.g. preheated) or other elements may be needed for engaging and for mechanically exerting stress in the desired forming operation.

Thereafter, treatment of the shaped article may be of any sort desired, essentially without regard to the fact that a condition of superplasticity has temporarily existed. Indeed such condition, including the stated instablity, will usually disappear at once, more or less simultaneously with the completion of forming or very briefly thereafter. The piece can be allowed to cool slowly or rapidly and can be subjected to heat treatments or other operations as may be conventionally desirable for the ultimate service of the article and for agreement with the metallurgical properties of the selected alloy. Indeed it is found that in a number of cases the resulting article has properties superior to those of articles formed by conventional procedure (such as repeated cold working and annealing), in being characterized by a, better toughness, smoother or otherwise improved surface quality, and indeed in the case of stainless steel, a lessening or avoidance of the surface shape characterized as roping.

If the nature of the required fabricationn is such that a single deformation is insufficient, it will be understood that the piece, after cooling, can be further worked in the same manner, as by again rapidly heating and holding at the temperature of microstructural instability, while a second deforming operation is performed, which may, of course, be similar to or different from the first. While in such repetition or repetitions it may not be possible to take advantage of recrystallization effects from a cold worked state, substantial superplasticity is nevertheless attainable.

Although the invention is not dependent on theoretical considerations, it is believed that the superplasticity is at least in substantial part occasioned by the change in structure (while it occurs) of some of the material from the body-centered cubic structure of alpha iron, which is the low temperature phase, to the face-centered cubic structure of gamma iron, which is the high temperature phase. Thus there is a driving force, so to speak, which produces such transformation and which may be deemed to contribute to the severity of instability. At the same time where the material has been heavily cold worked, the concurrent recrystallization affords a like driving force in converting the elongated crystals to very small equiaxed ones. Indeed tests have been made by quenching the deformed product immediately after such deformation, and examination of the microstructure has indicated a finer ferrite grain, including a coarse substructure as discussed below.

It is also believed that even where there has been no previous cold working, the deformation, during transformation instability, may be accompanied by a so-called dynamic recovery, which enhances the unstable condition. The preference for operation in the two-phase field is that the instability is there considerably prolonged, so as to afford full opportunity for deformation time and for avoidance of difficulty close control of time and temperature. Indeed it may be noted that exact treatment temperature values do not usually have to be maintained, e.g. in the sense that if the theoretical optimum is 1,500°F., the selected temperature can actually be in a range, as from 1,475° to 1,550° F.

The invention is applicable, as stated above, to a variety of steels, which are normally non-austenitic, i.e. which are not austenitic at room temperature as commonly processed, and especially in such steels having a two-phase region of austenite plus ferrite at specific elevated temperatures. These include, in addition to the ordinary carbon steels (most advantageously up to about 0.5% C), any of a wide variety of alloy steels, of which a few examples of compositions that are appropriate for making articles of the sort for which the invention is especially suitable, are grades 4340, 8620 and 10B20, the latter being a boron steel corresponding to grade 1020. It is likewise conceived that a number of high alloy grades can be treated. Particular utility is contemplated for stainless grades of the 400 series as mentioned above, these being generally straight chromium compositions, with chromium content ranging from 11 to 27%, and generally lacking nickel as an intended alloying element, except for one or two grades with nickel up to 2.5%. Specific examples of stainless steel are AISI types 409, 410, 430, 434 and 436, which have carbon contents up to about 0.2%. All of the above steels, whether of ordinary, alloy or stainless types, and including those which are conventionally classed as martensitic, may be generically deemed to be normally ferritic compositional character.

The forming operations conceived as useful include drawing and stretching, pressing, coining and similar die procedures, and indeed a variety of operations often attempted in the cold state, such as those of the nature of heading, upsetting, and like impact methods. Not only is there obtainable a very large extent of deformation, preferably upwards of 100% in uniform elongation, but a letter conformity with intricate die configurations, thus permitting single step fabrications in many cases not heretofore attained. Indeed parts or shapes which could not at all be produced in one piece may now be so made. As also stated, suitably high strain rates are realized, well above one or preferably

10 per minute, and indeed up to 1,000 per minute or higher if feasible.

A further % of the invention is represented by application of the process to stainless steel of the 400 series, i.e. so-called straight chromium compositions, particularly stainless steel type 430 having a nominal chromium content of 145 to 18%. For these tests a commercially produced 430 grade stainless steel was used, containing, by weight, chromium 16.25% and carbon 0.073%, with the usual maximum limits for incidental elements (e.g. Mn and Si each under 1%).

The tests were performed in essentially the same way as in the other examples above. The steel had been produced by ordinary processing, including hot rolling to appropriate plate thickness. A part of this plate material was subjected to cold rolling to a reduction of 60% (without subsequent annealing), and another portion was left in the as-received condition, i.e. with no further work after hot rolling. Cylindrical specimens of both types of material were prepared, ¼ inch in diameter and 7½ inches long, providing a grip separation of 4½ inches. The same test equipment was employed as in preceding examples, having provision for heating the specimen rapidly to a selected temperature, and then allowing it to be held at such temperature while deformation was effected, i.e. by stretching. The desired determination was with respect to superplastic conditions, as represented by stretch deformation to a maximum reduction of cross-section before occurrence of necking. In particular, the tests regarded as significant for this steel, within the competence of the equipment described above, were measurements of uniform reduction in area at various test temperatures, and likewise the deformation loads required, i.e. stresses needed to deform the work piece.

The results of the tests, illustrating the effectiveness of the invention in this type of stainless steel, are shown in FIGS. **13** and **14.** Each plotted point represents a separate test, being the same test for the two figures, wherein a specimen was subjected to rapid heating, was held at the indicated temperature and was promptly deformed while at such temperature and specifically, while it remained in such state of microstructural instability, if any, as occurred. In each case the specimen was heated at a rate of about 125° F. per second, requiring a time of about 12.5 seconds to reach the test temperatures in the optimum localities of the range. The specimen was held for 10 seconds at the selected temperature and then immediately subjected to deformation by axial stretching, while remaining st temperature, at a cross head speed of 90 inches per minute. In each of the figures, the plotted triangles represent the as-received specimens, with no cold working, and the circles represent specimens which were characterized by 60% cold reduction.

As illustrated in FIG. **13,** the tests showed that when the specimens were heated to a temperature in the range of about 1,470° F. to about 2,100° F., they exhibited highly desirable values of e_D as defined above, particularly values of the order of 0.4 or better, indicating deformation with uniform reduction in area, and no necking, to elongation of about 50% or higher, in contrast to normal uniform elongation for 430 stainless steel, of not more than about 20%. It was evident that at these temperatures the metal was temporarily in a condition of microstructural instability while alpha to gamma transition was occurring.

The cold worked specimens showed significantly higher values of e_D, understood to result from the contribution of the occurrence of recrystallization to the total microstructural instability that afforded the superplastic state. FIG. **13** expressly shows that the increase in ductility was roughly three-fold from the situation of specimens at 1,200° F., which is understood to be approximately the same as at room temperature. It is also understood that if the microstructural instability were not present, i.e. as by holding the metal for a long time at a given temperature so that the microstructure was stabilized before deformation, the values of e_D would be represented approximately by the dotted line curve in FIG. **13.**

FIG. **14** shows the values of maximum load required to deform the specimens at the selected temperatures. As in the case of other steels mentioned above, a drastic decrease in deformation load requirement was exhibited over the temperature range of superplasticity, including a three-fold decrease between 1,200° F. and 1,580° F., thereby further substantiating the applicability of the invention to the steel, in attainment of this additional advantageous property. Strain rates, as will be understood, were well within the preferred ranges indicated above, the test operations being at the same cross head speed as in the other examples.

It will be noted in FIG. **13** that the improved elongation characteristics were found over a plateau of temperatures from about 1,470° F. to 2,100° F., and rose substantially at much higher temperatures. Similarly the deformation load requirements decreased, as shown in FIG. **14,** over the plateau temperature range indicated in FIG. **13** and beyond. It will be understood, however, that the major significance of these tests is the improvement experienced in the lower temperature values of the plateau range, particularly in that higher temperatures are less commercially desirable, and indeed at exceptionally high values would become very difficult to utilize. At the same time, increases of temperature above about 1,580° F. do not significantly improve the ductility until values of about 2,100° F. are reached. In consequence optimum temperatures for the selected steel are represented by the range of about 1,500° to about 1,700° F., preferably 1,550° to 1,600° F.

The above tests, as illustrated in FIGS. **13** and **14,** afford abundant confirmation that the invention is applicable to alloy steels such as the stainless grades of the 400 series and that the described, large increase in ductility or superplasticity is attained for a brief but sufficient interval when the steel has been heated rapidly to a convenient temperature in the alpha-gamma phase transformation range, with the aid of concomitantly occurring recrystallization when the metal has been cold worked. The difference in nature of results at very high temperatures, and thus of the shapes of the curves in FIGS. **13** and **14,** from those for example in FIGS. **2, 3, 7, 8** and **11,** is understood to be accounted for by the different characteristics of the alloy compositions, as indicated by a typical example of a phase diagram for these stainless grades, shown in simplified form in FIG. **15.** In this graph, which is essentially a plot of equilibrium phase conditions various temperatures, for various weight percent contents of chromium, and which for convenience of illustration is specifically such a plot for alloys containing 0.05% carbon (it being understood that generally similar configurations pertain to

other carbon levels, with specific differences in shape and location of phase boundary lines), it will be seen that there is a so-called gamma loop as indicated at **20.**

More particularly, in FIG. **15** the gamma loop is surrounded, in effect, by an alpha plus gamma band **21,** representing a region for occurrence of the same kind of transition as is involved in the triangular area of FIG. **12.** Regions below these areas **20** and **21,** e.g. as generally indicated at **22,** can be considered equivalent to the alpha or alpha-cementite regions at the bottom of FIG. **12,** i.e. where the ferritic type of microstructure prevails, as distinguished from the basic austenitic structure within the gamma loop **20.** It will be understood that the upper portion of the complete diagram, where the boundaries of regions **20** and **21** return to the zero chromium axis, is omitted from FIG. **15** as being of no concern here, and likewise certain special characteristics are omitted relative to regions below the band **21,** or partly within it, where, depending on temperature and chromium content, there are varying proportions of one or more iron-chromium carbides. For simplicity the latter features of composition have been merely indicated by the letter "K," it being understoood that at various localities, "K" comprises one or more of various iron-chromium carbide compositions.

As will be recognized from phase diagrams of the sort shown in FIG. **15,** ferrite-austenite transition regions can exist over a large range of temperatures for at least some alloys, thus presumably accounting for the phenomena at very high temperatures in FIGS. **13** and **14,** while nevertheless affording practice of the invention at conveniently lower temperatures for all these alloys, as explained. Moreover, although the equilibrium phase diagrams, including the position of the gamma loop, vary for different alloys of these types and specifically for different carbon levels, and the phase boundaries may also shift somewhat with heating rate, and although a complete transformation to austenite may not be attainable, alpha-gamma transition regions appropriate for present purposes are available in generally all cases, e.g. for the variations in chromium content in the 400 series and for variations in carbon level, from extremely low values up to 0.5% or higher. In other words, while the driving force for austenite transformation on rapid heating into the two-phase field is more complicated in these alloys than for ordinary carbon steel, this circumstance does not alter the basic applicability of the present invention.

As indicated, these results demonstrate the attainment of the desired superplastic state with the ferritic grades of stainless steel, it being understood (as explained above) that the term ferritic or "normally ferritic" is herein employed to include those particular grades which are usually martensitic in the as-produced cold state, in that the reversible ferrite-martensite transformation occurs at lower temperatures than the terminal values of the present process, which consequently involves rapid heating, in all cases, upward through the ferrite region to the point of alpha-gamma transition. As shown by FIG. **13,** advantage may be taken of the instability of occurring recrystallization in these stainless steels in the same general way as for the plain carbon grades. The extent of cold working required to effectuate this contribution to high ductility appears to vary with steel compositions, but it will be understood that the fact of such contribution and the percentage of cold work needed can be readily determined by tests of the sort illustrated in FIG. **13,** for any given steel. In other words, although the advantageous effects of recrystallization are not presently deemed essential (where the instability by phase transition can occur), they appear to be generally capable of determination and realization in desired cases.

The products of the invention resulting from significant deformation while the steel is held in the temporary state of microstructure instability have been found to possess mechanical properties, especially as to toughness or impact strength, and also as to yield and tensile strength, superior to the original steel. The toughness, moreover, can be very much better than would characterize the same steel not treated by the process but instead directly subjected to cold deformation.

These results have been demonstrated by tests, of which representative examples are here set forth. The material employed was a steel hot rolled to plate, e.g. such as employed as skelp for making welded pipe, having a thickness of ½ inch and a composition as follows: 0.1% C. 0.95% Mn, 0.06% Cb, 0.04% Al (aluminum killed), rare earth additions (less than 0.05% total), and incidental elements, including very low sulfur (0.006%) and silicon below 0.01%. This was a commercial skelp material, which can be considered a substantially non-alloy carbon steel, available under the identification X–52, and the three plates used had yield strengths in the range from 57,000 to 66,000 psi. Massive specimens, 0.5 × 0.5 × 7 inches, were prepared and used in the above described Gleeble apparatus. In each instance of treatment of a specimen it was heated rapidly to one or another of three selected temperatures, viz. 1,350°, 1,450° or 1,550° F., i.e. at a rate faster than 10° F. per second, specifically at about 50° F. per second. Some of the specimens were held for 10 seconds at temperature and then (while fully superplastic) were deformed, i.e. by stretching, to approximately 20% reduction in area of cross section, uniformly along the axis of stretch with full advantage of the superplasticity. In other cases the specimen was held for 300 seconds and then similarly deformed. In each instance the deformed object was thereafter air cooled. Control specimens were subjected to the same thermal cycles without deformation.

From all specimens, both deformed and control, suitable test specimens were prepared, e.g. for hardness and impact testing, and also for measuring tensile properties as described further below. The specimens for Charpy toughness (impact tests) were prepared, for convenience, at two-thirds of the usual size, but will be understood that the comparative results obtained are fully significant, since all tests were made at the reduced size.

Test specimens were also prepared from the plate as received, from the same received material which was reduced 10 to 15% in thickness by cold forging.

The hardness tests showed that there was no significant deterioration in hardness by the procedure wherein the steel was deformed in superplastic state, especially in the test of material which had been deformed respectively at 1,350° and 1,450°. The specimens remained in the Rockwell B hardness range of 80–90, whether measured with respect to the transverse or longitudinal orientation, relative to the direction in which the received plate had been hot rolled. When the deformation was effected at 1,550° F., the

hardness departed to or a little below the lower end of the above mentioned range. There was some variation among various specimens, but results clearly demonstrated that the product of the high temperature deformation during microstructural instability, i.e. the ultimate cooled product, maintained good hardness.

The Charpy impact tests showed very substantial improvement in impact strength, especially in respect to the steel which had been deformed while maintained in the upper superplastic state at 1,450° or 1,550°. As is conventional in toughness testing, readings were made of the prepared V-notched test bars, while held at room temperatures, i.e. 70° F. and also at a suitably cooled temperature, −20° F., the measurements being in foot pounds of energy required to break the bar. These tests were made with specimens oriented both longitudinally and transversely as to the original direction of hot rolling of the plate. The same original plate material, untreated by the process, showed Charpy values of 60–67 pounds at 70° F. (and 4–16 at −20°), in the longitudinal direction. After holding 10 seconds at 1,350° and deforming to an area reduction of 17–22%, the Charpy values at 70°, longitudinal, were 68–74 foot pounds. When the deformation was effected at 1,450°, e.g. for 24% reduction in area, these longitudinal values were 86–95 foot pounds. At 1,550° with similar treatment effecting reduction of approximately 15%, the ultimately cooled-down specimens, tested for impact, yielded values of approximately 118 foot pounds at 70°.

In the situation of longitudinal Charpy measurements for the low temperatures, the material treated at 1,350° showed essentially no improvement, but at 1,450° there was very substantial improvement in the longitudinal direction. Thus where reduction in area had been 17.6%, tests at −20° F. showed that the Charpy value was 75 foot pounds and where the reduction was 23.8, the impact value was 57 foot pounds. This large improvement was also exhibited in the material which had been formed at 1,550°, the results, for 15–18% reduction being 100–105 foot pounds at −20°, longitudinally.

In the transverse direction, the nature of the results was similar to that in the longitudinal direction, in that the impact or toughness values improved significantly, especially where the material had been deformed at 1,450° and also at 1,550°, there being also some improvement in the +70° readings for product that had been reduced during holding at 1,350°. Whereas specifically, in tests at 70°, the original material showed impact values (transverse) of about 47 foot pounds, the values after the 1,450° treatment (10 second holding) were 62 and 68 foot pounds respectively for material reduced 25 and 10.6%. For the material processed at 1,550°, with reduction of 15–18%, the Charpy impact values (transverse) at 70° were about 75 foot pounds. In the measurements at −20° F., there was a correspondingly substantial improvement in toughness, e.g. in the transverse direction. Whereas the original material had Charpy values of 18.5 to 28.5 foot pounds at −20° (transverse), the product that had been processed at 1,450° with 18–22% reduction showed transverse impact values in the range of 34–43 foot pounds. Where the processing was at 1,550°, for a reduction of 14–17%, the impact value of the product at −20°, transverse, was approximately 61 foot pounds.

Results similar to those reported above were found in material that was processed and deformed at 1,450° and 1,550° with a holding time of 300 seconds before deformation.

It is noted that specimens which had been given the same thermal cycle, but without any deformation during the interval of temporary instability, also showed toughness improvement, but, of course, these specimens did not have the advantage of having been deformed with unusual facility to the desired, ultimate state.

Tests were also made to determine the effect on toughness of cold forging similar plate material, i.e. as distinguished from the deformation in accordance with the present invention. In these tests, the Charpy V-notch values decreased very markedly in both longitudinal and transverse directions, and at both +70° and −20° temperatures, when the material was cold reduced, by cold forging, to an area reduction in the range of above 10 to 15%. In one instance longitudinal value dropped from over 100 foot pounds to 40 foot pounds at 70° F. and from upwards of 70 foot pounds, down to 26 foot pounds or less at −20° F. In the transverse direction, the drop was from about 60 to 33 foot pounds, transversely, at 70° and from 40 foot pounds or more to about 10 pounds at −20°. These results emphasized the significance of the impact or toughness strength improvement achieved by the present invention, in the production of deformed steel articles. In other words, as distinguished from articles prepared by cold working, the deformation to a desired shape by the present process, yields products of very greatly superior toughness. Instead of a substantial decrease in this strength value, the new products have a large increase over the original, hot-rolled material. It will be appreciated that these operations permit the attainment of products having excellent properties, without special alloying or other special operations heretofore deemed necessary.

Upon practice of the defined process, with a significant deformation of the material, e.g. more than 5% reduction in area, or very preferably at least 10%, the steel product, as ultimately tested after air cooling, has unusually high properties, without loss in other respects. The deformation can be performed in any suitable manner, by drawing, forging, rolling or the like, and it will now be apparent that special uses of the invention are to obtain products that may not otherwise be obtainable, e.g. rolled high-strength, low-alloy steel at gauges smaller than heretofore possible with hot rolling, while avoiding the loss of impact strength and ductility that usually occurs in cold rolling. Similarly, steel can be rolled by this process to achieve precision bar products, of dimensional precision, with good strength, as contrasted to currently imprecise hot-rolled bar products that now must be further processed by cold drawing to achieve dimensional precision but in such case suffer loss of impact strength and ductility. As stated, the improved products of the invention are obtainable for a variety of steels, as explained above, and for a wide variety of deforming operations and ultimate shapes or configurations.

Suitable tensile test specimens were prepared from the specimens of X–52 steel which had been subjected to the present procedure and of which other pieces were used for impact tests. These tensile specimens were given standard tensile tests, determining ultimate tensile strength and also yield strength, which in the case of this steel could be measured at the upper and

lower points, i.e. upper and lower yield strengths. The tensile measurements were made with respect to the longitudinal and transverse aspects of the steel, for specimens which had been processed at the temperatures stated above, namely 1,350°, 1,450° and 1,550° F. Mostly the tests were related to the steel which had been held for ten seconds at the selected temperature and then deformed during microstructural instability, i.e. to reductions in area in the range of 11–24%.

These tests demonstrated that generally over the range of temperatures of deformation, the steel maintained its original tensile properties or became characterized by improved tensile properties — comparing untreated specimens with those which had been treated by the process and cooled as above described. Whereas the original material in longitudinal direction showed upper and lower yield strengths of approximately 57.5 ksi (thousands of pounds per square inch) and ultimate tensile strength of 74.5 ksi, the treated material showed roughly about the same values for deformation at 1,550° F., with somewhat better values at 1,450° F. and with still higher values at 1,350° F., i.e. ranging to lower and upper yield points of about 70 and 75 ksi and ultimate tensile strength of 83.5 ksi. At the intermediate temperature of 1,450° the three values were respectively about 66, 72 and 78 ksi.

In the transverse direction a similar situation was found, where the original material had upper and lower yield points of 65 ksi and ultimate tensile of 79 ksi. Here the material treated at 1,550° F. showed a lower yield point of about 64, upper yield point about 72 and ultimate tensile about 72 ksi. Likewise at 1,450°, in the transverse direction, the steel so treated showed lower yield, upper yield and ultimate tensile strength of about 69, 76 and 80 ksi respectively, while for the steel treated at 1,350° these values rose to about 74, 81 and 85 ksi. With increase of yield and ultimate strengths, the present elongation and percent reduction of area on yield decreased, but the properties in these respects remained satisfactory.

The tensile properties of the treated steel and the impact properties can be considered together in that, for example, over a treating range of 1,350° to 1,550° F., the impact properties are generally highest for highest temperature of treatment, whereas tensile properties are generally highest for the lowest temperatures in the range. Nevertheless, both the impact properties and the tensile properties are at least substantially equal to those of the original steel over the temperatures of the process. As a result, the product can be generally characterized as having superior properties in one or the other of these respects and at least as good properties in each respect, in comparison with the original body of steel. Moreover, it will now be seen that by selecting the temperatures of treatment, i.e. for development of superplasticity, the product can be produced to have a selected improvement. This can be either improvement in both impact and tensile properties, as with these steels processed at 1,450° F., or the product can be made to have greatly increased tensile properties with essentially the orginial toughness, or greatly improved impact properties with the orginal tensile strength, these selections being respectively at the lower and upper ends of the processsing temperature range.

Some other tests indicated that the hold time at the elevated temperature of instability (superplasticity) had some bearing on the ultimate properties of the deformed article, in the sense that increasing the hold time to 300 seconds tended to decrease the tensile properties slightly at the lower temperatures of treatment, while the longer hold times appeared to afford some further increase of impact strength.

Inasmuch as the yield strength values and the ultimate tensile values generally always varied in substantially the same way, i.e. as to increase or decrease or lack of change, with processing or changes of conditions, this area of mechanical properties can be simply identified as tensile strength for convenient identification herein.

Finally, it is found that the steels processed in accordance with the present invention are characterized by a distinctive microstructure, which is believed to be new and unique. FIGS. **16** and **17** are photomicrographs of sections of steels that have been processed according to the invention and are representative of the new products. Specifically, these are photographs taken respectively at 200X and 400X magnification of suitably prepared specimens, FIG. **16** being the 1006 steel, originally in an annealed state and deformed at 1,495° F., and FIG. **17** being the 1018 steel which had been cold worked 50% and which was deformed at 1,425° F. This new structure is understood to represent the ferrite grains, which now contain an unusual substructure, i.e. a coarse, well-defined cell structure within the grains, as shown in each of these views. The microstructure also includes carbon-rich or carbide-containing areas (dark etching areas in the photographs) identified as an austenite decomposition product.

The specimens employed, which are helium gas quenched from the above-quoted deformation temperatures, were processed and photographed by conventional methods, for revealing the ferrite grain structure. Standard metallographic techniques for specimen preparation, which include grinding and polishing, were employed. After etching in a nital solution, the specimens were examined using normal bright field optical illumination. As will be understood, the photomicrographs in FIGS. **16** and **17** show representative areas and were taken at the above-stated magnifications.

It is to be understood that the invention is not limited to the specific operations and materials herein described, but may be carried out in other ways without departure from its spirit.

We claim:

1. A steel product having a selected shape produced by deformation of a body of steel which has a compositional character that is normally ferritic and is such that as exhibited by the equilibrium phase diagram for said compositional character there is an alpha-gamma-transition temperature value capable of providing temporary microstructural instability, said steel product having substantially greater impact strength than said body of steel, said steel product being ferritic and having a microstructure composed essentially of ferrite grains characterized by a coarse cell substructure within each of the grains, and said steel product being produced by: subjecting said body of steel to rapid heating, at a rate of at least about **10°** F. per second, to the aforesaid temperature value, holding the body at said value for providing high ductility in said body during an interval of microstructural instability while transformation occurs in the steel from alpha iron at

least partially to gamma iron, and subjecting the body to rapid deformation to said selected shape by applying stress thereto while the body is held at the said temperature value in said interval.

2. A steel product as defined in claim **1,** in which the steel thereof is essentially a carbon standard steel having a carbon content in the range up to about 1%.

3. A steel product as defined in claim **1,** in which the steel thereof has a carbon content in the range up to about 0.5%.

4. A steel product having a selected shape produced by deformation of a body of steel which has a compositional character containing less than 0.8% carbon, that is normally ferritic and is such that it exhibits a phase field, in a range of elevated temperature values, which at equilibrium contains both ferrite and austenite, said steel product having substantially greater impact strength than said body of steel, said steel product being ferritic and having a microstructure composed essentially of ferrite grains characterized by a coarse cell substructure within each of the grains, and said steel product being produced by: subjecting said body of steel to rapid heating, at a rate of at least about 10° F. per second, to a temperature in the aforesaid range, such that upon reaching such temperature the steel experiences temporary microstructural instability, while phase transformation is occuring, and subjecting the body to rapid deformation to said selected shape by applying stress thereto while the body is undergoing said instability at said temperature.

5. A steel product as defined in claim **4,** in which the steel thereof is essentially a carbon standard steel of hypoeutectoid character having a carbon content in the range up to about 0.5%.

6. A steel product as defined in claim **5,** in which the temperature to which the body of steel is heated is about 1,450° F.

7. A steel product having a selected shape produced by deformation of a body of steel which has a compositional character that is normally ferritic and is such that as exhibited by the equilibrium phase diagram for said compositional character there is an alpha-gamma-transition temperature value capable of providing temporary microstructural instability, said steel product having substantially greater strength in at least one of the properties of impact strength and tensile strength than, and at least as great strength in each of said properties as, said body of steel, said steel product being ferritic and having a microstructure composed essentially of ferrite grains characterized by a coarse cell substructure within each of the grains, and said steel product being produced by: subjecting said body of steel to rapid heating, at a rate of at least about 10°F. per second, to the aforesaid temperature value, holding the body at said value for providing high ductility in said body during an interval of microstructural instability while transformation occurs in the steel from alpha iron at least partially to gamma iron, and subjecting the body to rapid deformation to said selected shape by applying stress thereto while the body is held at the said temperature value in said interval.

8. A steel product as defined in claim **7,** in which the steel thereof is essentially a non-alloy carbon steel having a carbon content in the range under 0.8%.

9. A steel product as defined in claim **7,** in which the steel thereof has a carbon content in the range up to about 0.5%.

10. A steel product as defined in claim **7,** in which the steel thereof is essentially a non-alloy carbon steel of hypoeutectoid character having a carbon content in the range up to about 0.5%.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,857,741 Dated Dec. 31, 1974

Inventor(s) Frank A. Hultgren, et al

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

The term of this patent subsequent to March 27, 1990, has been disclaimed.

Signed and Sealed this

Seventeenth Day of August 1976

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks