Dec. 7, 1954 W. R. BREELER 2,696,450

AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Filed July 22, 1952 7 Sheets-Sheet 1

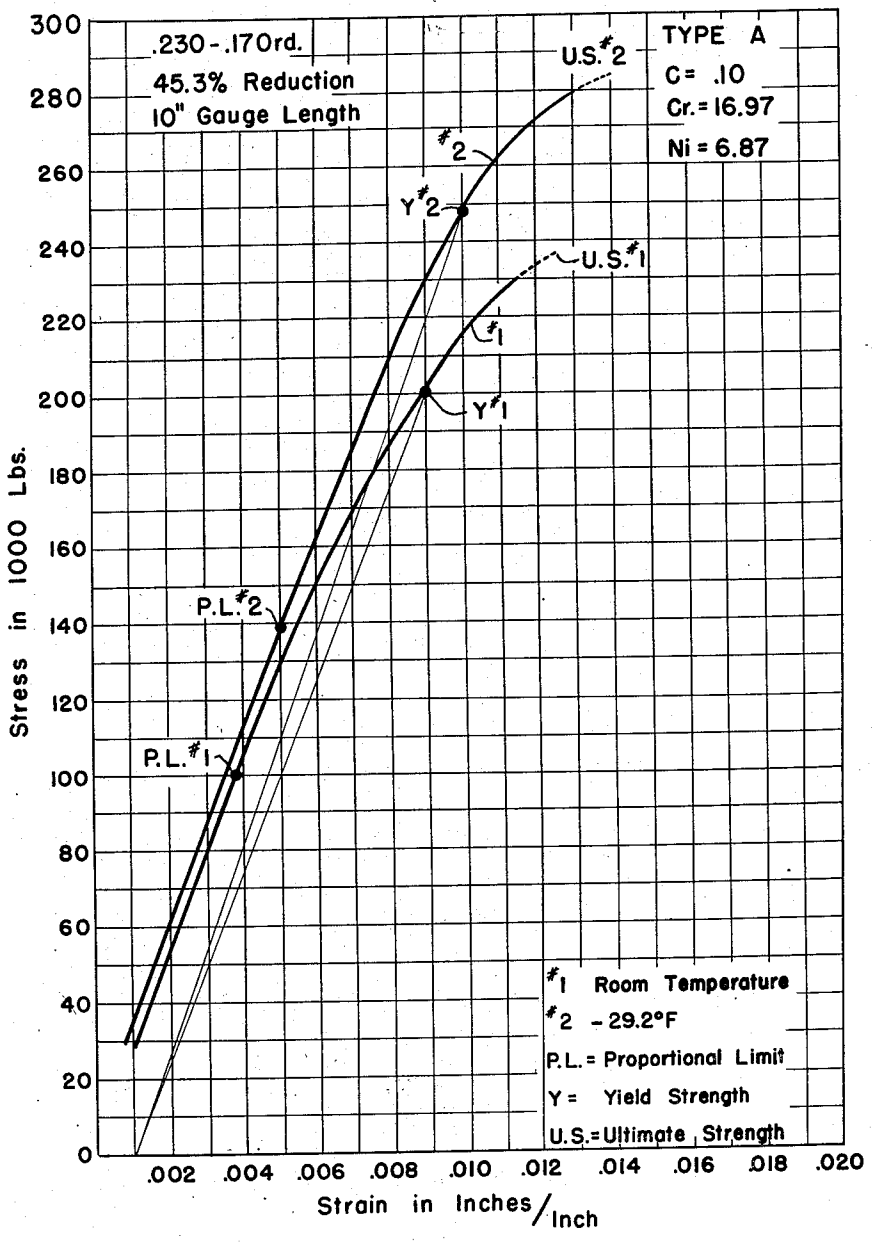



Fig. 1

INVENTOR.
Walter R. Breeler
BY Green, McCallister & Miller
HIS ATTORNEYS

AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Filed July 22, 1952 7 Sheets-Sheet 2

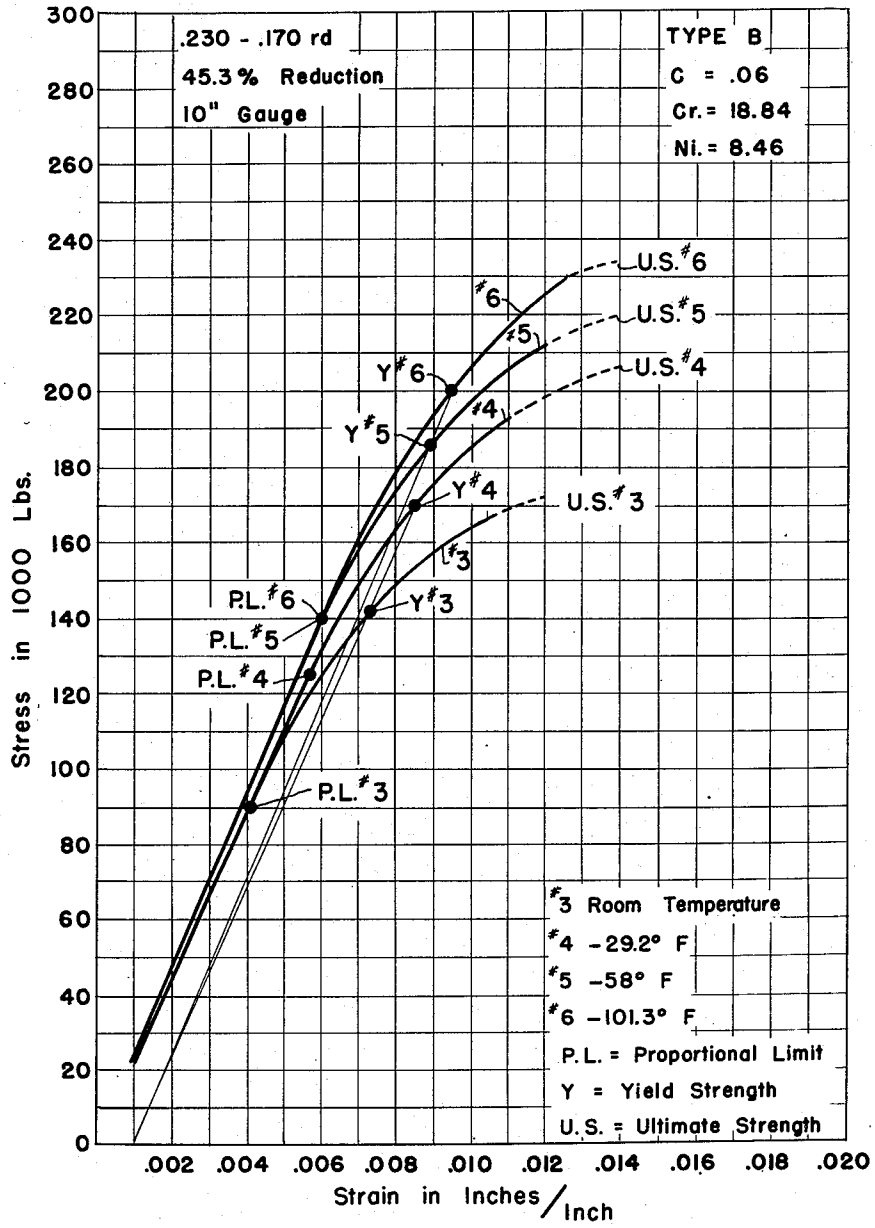



Fig. 2

INVENTOR.
Walter R. Breeler
BY Green, McCallister & Miller
HIS ATTORNEYS

Dec. 7, 1954 W. R. BREELER 2,696,450

AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Filed July 22, 1952 7 Sheets-Sheet 3

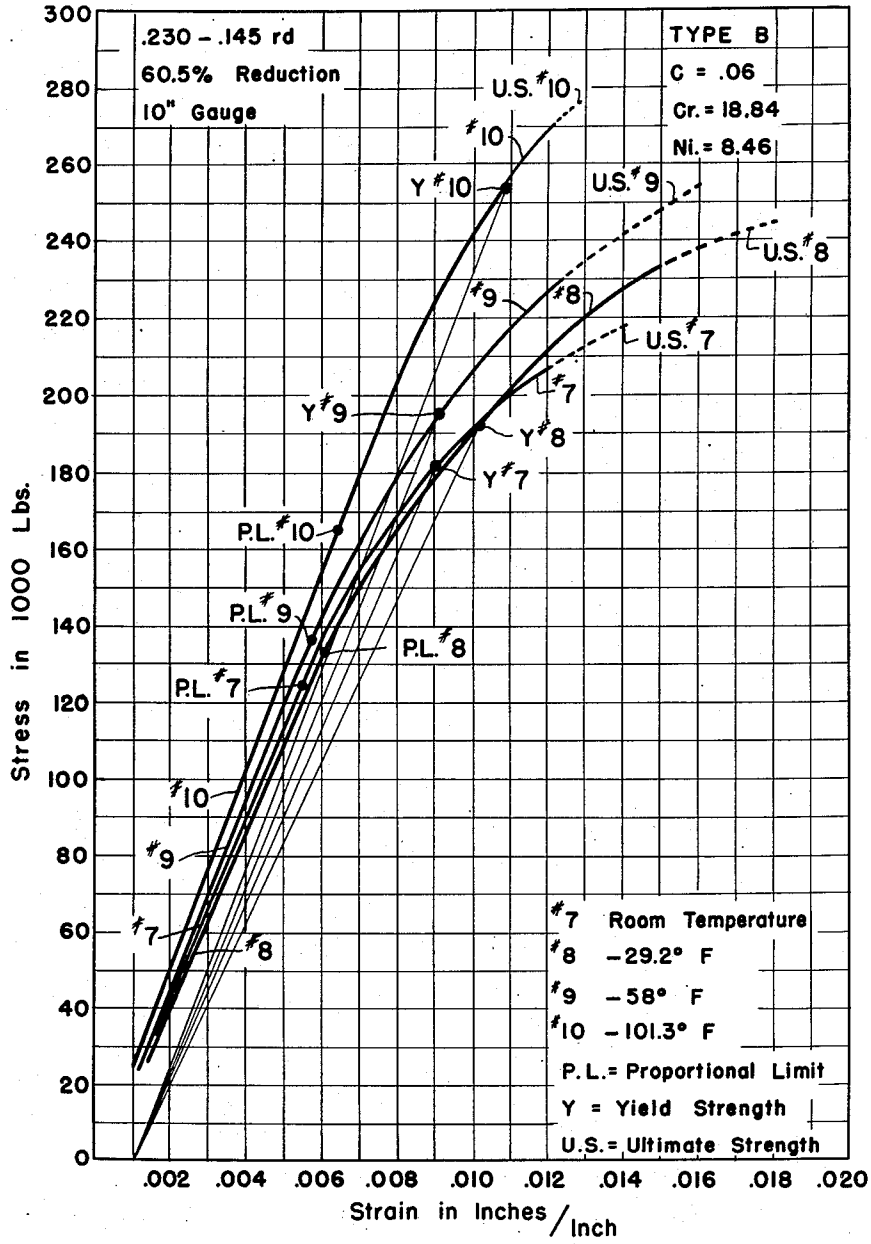



Fig. 3

INVENTOR.
Walter R. Breeler
BY Green, McCallister & Miller
HIS ATTORNEYS

AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Filed July 22, 1952 7 Sheets-Sheet 4

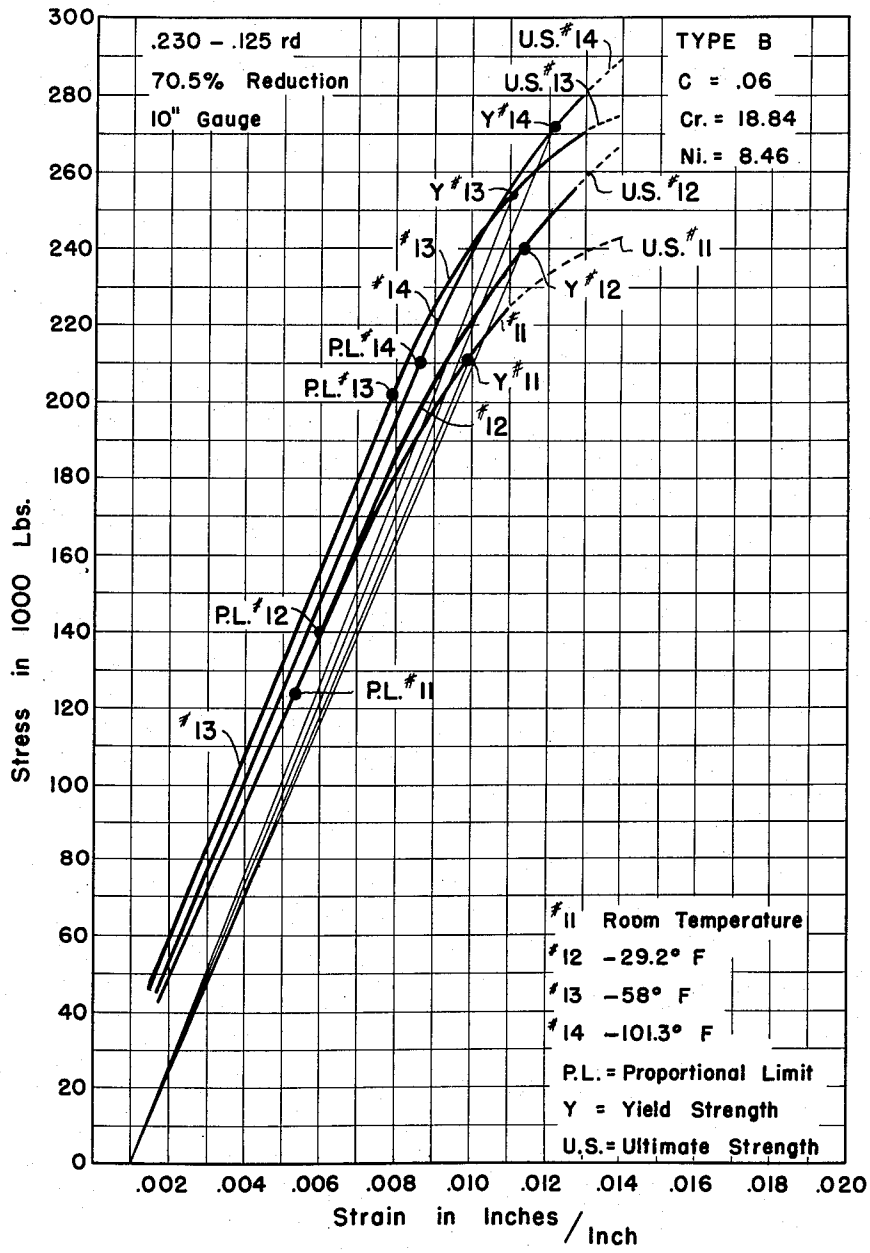



Fig. 4

INVENTOR.
Walter R. Breeler
BY Green, McCallister & Miller
HIS ATTORNEYS

AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Filed July 22, 1952 7 Sheets-Sheet 5

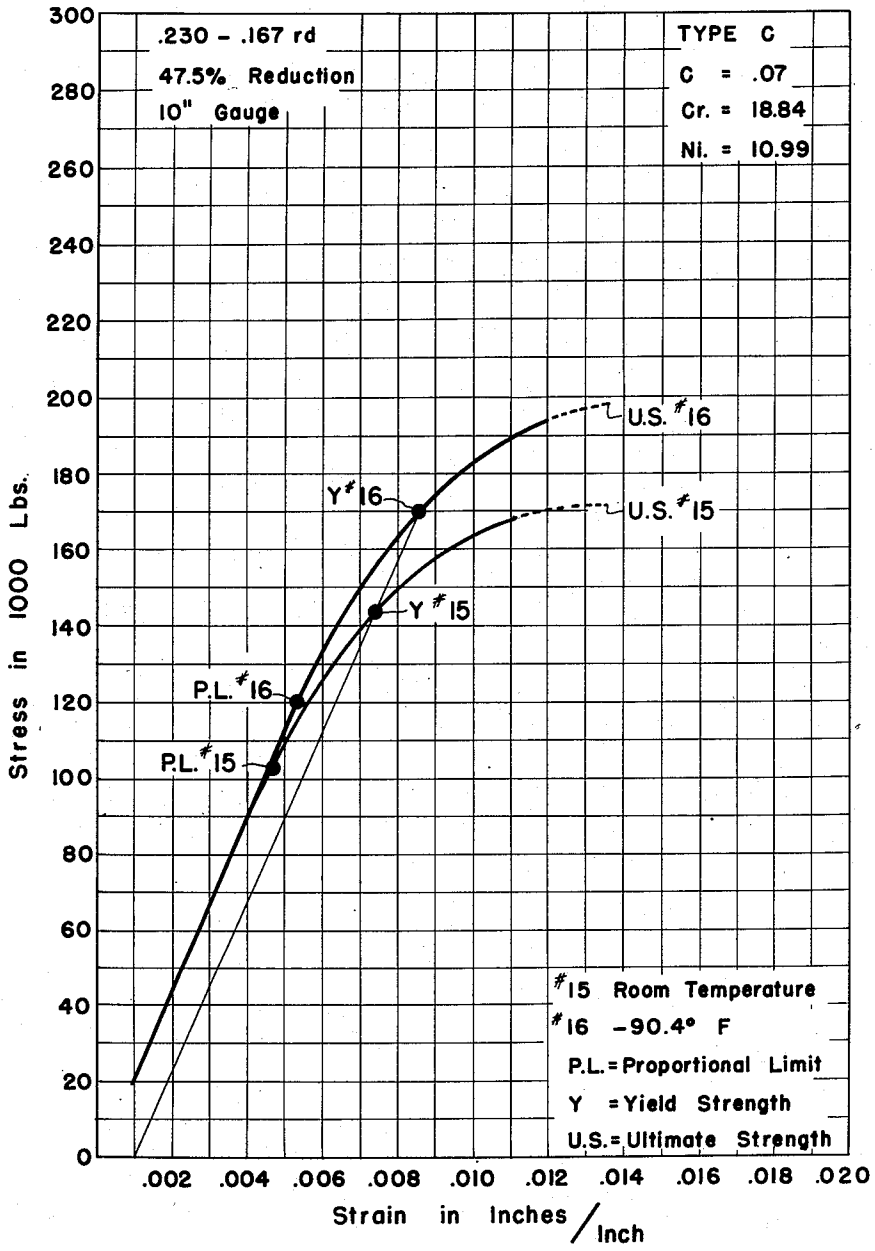



Fig. 5

INVENTOR.
Walter R. Breeler
BY Green, McCallister & Miller
HIS ATTORNEYS

AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Filed July 22, 1952 7 Sheets-Sheet 6

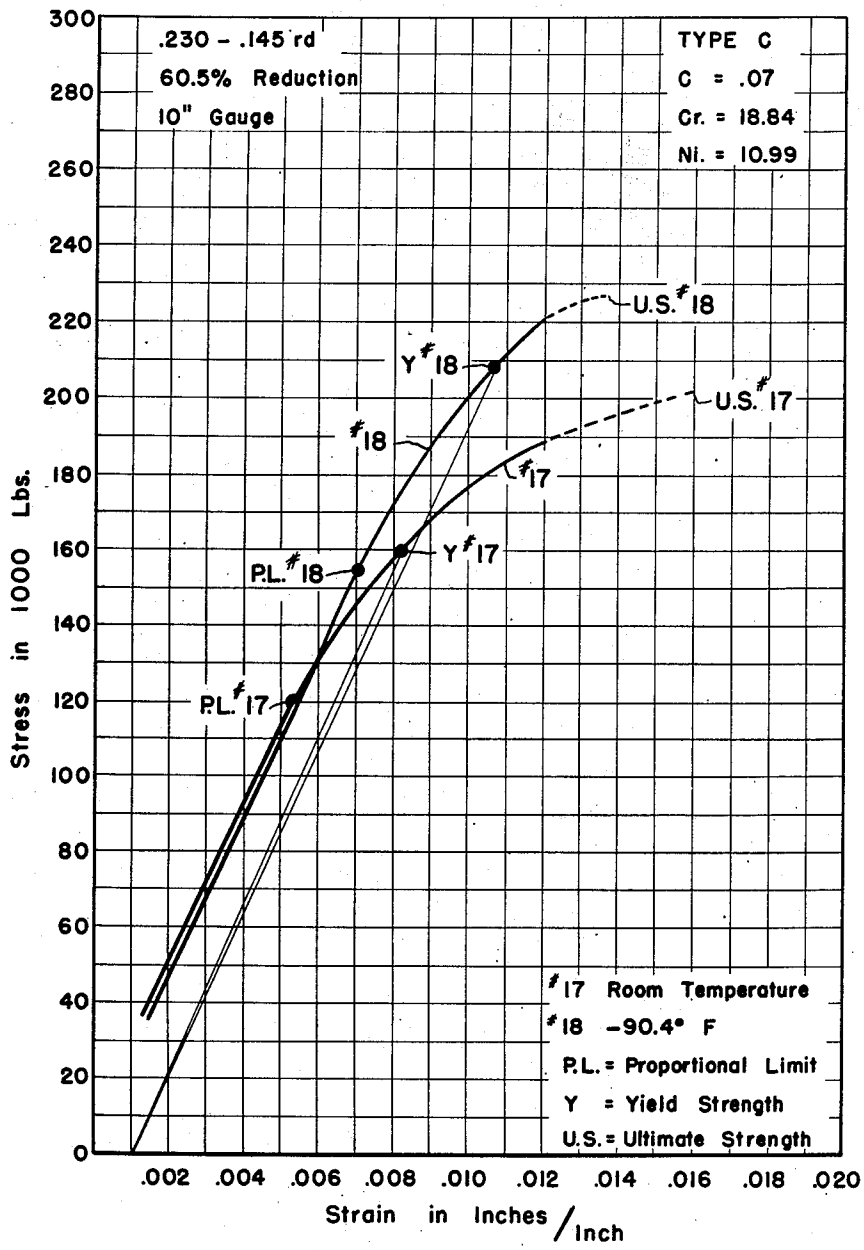



Fig. 6

INVENTOR.
Walter R. Breeler
BY Green, McCallister & Miller
HIS ATTORNEYS

AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Filed July 22, 1952 7 Sheets-Sheet 7

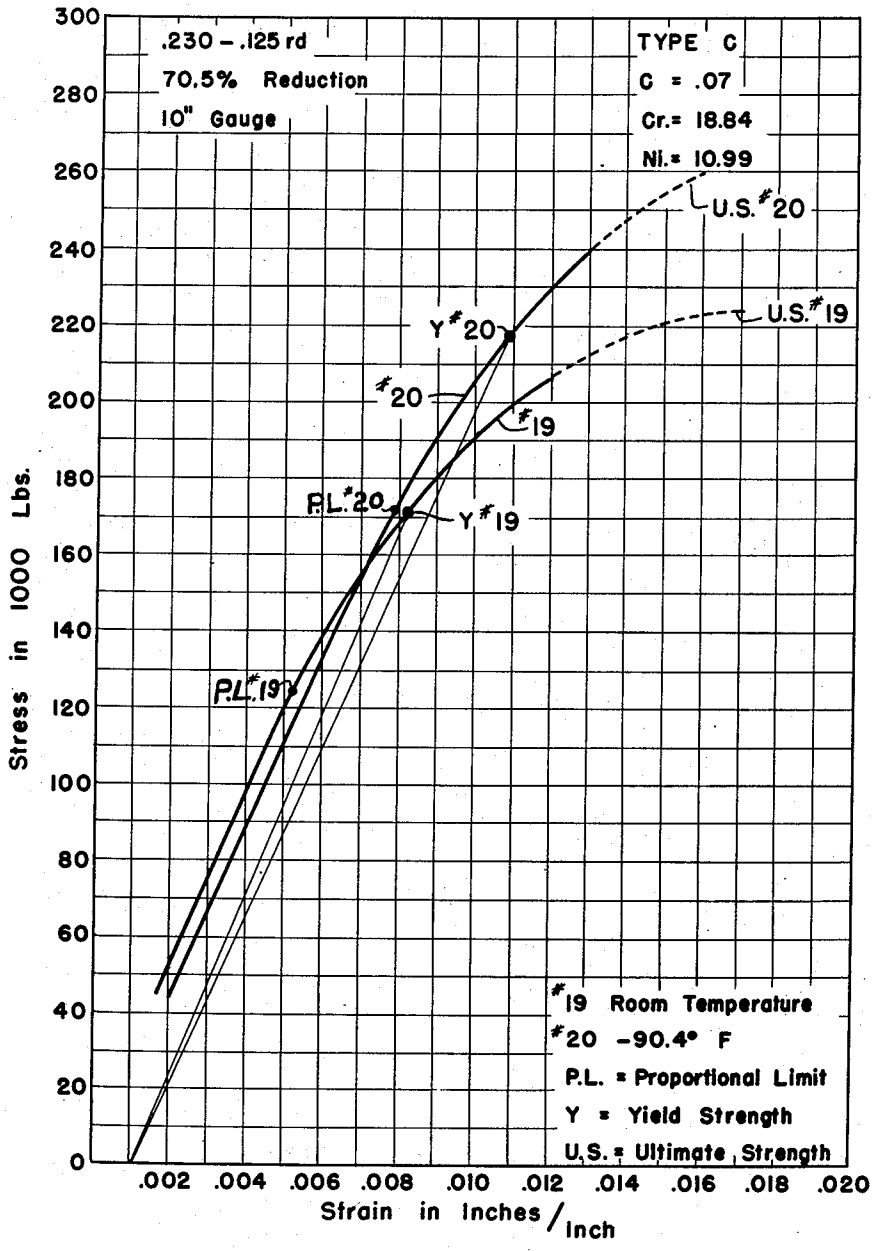



Fig. 7

INVENTOR.
Walter R. Breeler
BY Green, McCallister & Miller
HIS ATTORNEYS

United States Patent Office

2,696,450

Patented Dec. 7, 1954



2,696,450

# AUSTENITIC ALLOY STEEL AND PROCEDURE FOR MAKING SAME

Walter R. Breeler, Fredonia, N. Y., assignor to Allegheny Ludlum Steel Corporation, Brackenridge, Pa., a corporation of Pennsylvania

Application July 22, 1952, Serial No. 300,227

8 Claims. (Cl. 148—12)

This invention relates to austenitic ferrous alloys of improved characteristics and particularly, to new and improved procedure for processing austenitic steels to obtain improved physicals.

An important phase of my invention relates to discoveries made in connection with the processing of austenitic steels which involves the essential employment of an initial subzero temperature treatment. The procedure is at least in part based upon novel discoveries made in connection with critical, low temperature-sensitive characteristics of the steel.

It is well known in the art that steels change their inherent structure when heated up to a temperature above a normal room temperature and when thereafter brought down to a normal room temperature. In such a case, the ultimate structure of the steel at normal room temperatures may be determined by the particular heat treatment involved. For example, it is known that at a relatively high temperature, there is a tendency for iron carbide which is nearly insoluble in iron at a normal temperature to form a solid solution with gamma iron and provide austenitic characteristics. It is also known that steels may be made austenitic at room temperatures by a proper proportioning of their chemical compositions and particularly, by the utilization of an essential austenitic former, such as nickel, the use of other austenitic formers such as manganese, the proportioning of austenitic enhancers each as copper, manganese, carbon and nitrogen with nickel, and limiting the amount of ferrite formers such as molybdenum, titanium, columbium, etc.

Although tensile strength, yield strength, etc., of an austenitic steel tend to increase at subzero temperatures (in this connection, I have reference to temperatures below zero °F.), I have found that the steel is in a condition that precludes fully plastically or compression-deforming it at such temperatures to develop or stabilize any improvement in physicals present at such temperatures. I have found that yield strength and tensile strength of the steel at such temperatures tend to closely approach each other, and if an attempt is made to fully plastically deform the steel, that the ultimate strength may be exceeded with resultant breakage. Thus, a definite problem is presented in endeavoring to, as a minimum, at least stabilize any improvement in properties that is present at such subzero temperatures and to do so, without damaging the steel and to provide a usable product. I have found the problem presented to be three-fold: (1) to so condition the steel as to at least not decrease the spread between its yield and tensile strengths and at the same time preliminarily fix its subzero temperature physicals; (2) to further and fully develop the structure of the steel and stabilize the developed structure without damage to the steel; (3) and to develop uniform and the best possible improvement of physicals in the steel.

One investigator has shot-blasted steel at a subzero temperature to surface forge it, followed by age heat treating it at 500° to 1000° F. (750° F. optimum), all to provide it with an increased hardness. Although, surface working at such temperatures appears to provide a surface hardness which is retained when the steel is returned to a room temperature and which is stated to be further improved by the aging heat treatment, a full and uniform improvement of its physicals is lacking and any improvement is limited to its hardened surface. There is a definite need for a practical procedure which will enable the steel, after an initial subzero temperature treatment, to be fully plastically deformed through its full thickness section to fully develop its subzero physicals without, at the same time, setting up a dangerous brittleness aspect, and in general, without introducing limiting factors incident to an attempt to so deform the steel at a subzero temperature.



It has thus been an object of my invention to provide a solution to the problem presented and to devise a commercially practical procedure utilizing subzero-temperature-treated austenitic steel;

Another object has been to provide a new and improved austenitic steel from the standpoint of its physical structure;

A further object has been to develop a procedure for practically and effectively deforming subzero-temperature-treated austenitic steel to obtain highly improved physicals;

A still further object has been to develop criteria for processing austenitic steels from an initial subzero temperature treatment;

These and many other objects of my invention will appear to those skilled in the art from the detailed description thereof and the date hereinafter set forth.

In the drawings, Figures 1 to 7, inclusive, represent strain-stress curves plotted for austenitic steels of different chemical composition, different percentages of full plastic work deformation (reduction), and at different initiating temperatures. In the curves of each figure, I have compared the strain-stress characteristics of conventionally cold (room temperature) deformed steels with steels processed in accordance with my present invention having the same chemical composition and subject to the same amount of work deformation, but the treatment of which is initiated at a specified subzero temperature.

More specifically, Figure 1 shows comparative curves for a somewhat unstable austenitic steel when given about 45.3% deformation work treatment;

Figure 2 shows comparative curves of a steel of intermediate stability subjected to about 45.3% deformation work treatment;

Figure 3 shows comparative curves for the steel of Figure 2, but subjected to about 60.5% work deformation and Figure 4 for the same steel as subjected to about 70.5% work deformation;

Figure 5 shows comparative curves of a still more stable steel subjected to about 47.5% work deformation, Figure 6 shows curves for the same steel as subjected to about 60.5% treatment, and Figure 7 shows curves for the same steel as subjected to about 70.5% treatment.

For the purpose of further reference, the alloy of Figure 1 will be termed alloy A, the alloy of Figures 2, 3 and 4 will be termed alloy B, and the alloy of Figures 5, 6 and 7 will be termed alloy C.

Although I have determined that austenitic steels in general may be processed in accordance with my invention, optimum results are obtained with an austenitic steel of intermediate stability which is initially predominantly austenitic in its characteristics at normal or room temperatures. Also, my procedure has been applied to previously annealed as well as previously cold

worked austenitic steels. For example, I have started with an austenitic steel as reduced by hot working and then applied my intermediate treatment instead of a conventional cold rolling, forging, upsetting, or drawing process.

Briefly stated, in carrying out my invention, I, (1) condition the austenite of an austenitic steel at a subzero temperature; (2) heat fix the conditioned austenite; (3) and finally, develop the conditioned structure of the steel to provide it with highly improved physicals. That is, I first cool the steel down to a temperature of not greater that 0° F., that may be as low as —320° F. and as an optimum, is within a range of about —30° F. and —120° F. After the steel (which may be in the form of bars, wire, strips, or sheets, for example) has been fully brought down to such a subzero temperature, it is then quickly (approaching instantaneously) brought up, by what may be termed a heat fix, to a higher temperature that as a minimum is 2 or 3 degrees agove 0° F. and as a maximum, up to about 350° F. (well below its ageing temperature). I have discovered that is highly essential that the steel be brought up at least slightly above 0° F. before it is plastically deformed or pressure reduced. This second step lowers the compression resistance of the steel to deformation or working and tends to slightly increase the spread between its tensile strength and yield strength without a further development of and without an appreciable change in its subzero physicals.

I have found that it is also essential that the steel be provided with a temperature by the second step of my process which will allow for any heating up of the steel incident to its essential subsequent deformation in accordance with a third step of my process. That is, if for example, a number of subsequent draw reductions the employed. the steel should not reach a temperature higher than 350° F. in the first reduction. If, on the other hand, the subzero temperature treated steel is permitted to very slowly come back or heat up to a normal or room temperature, it will return to its previous, substantially stable austenitic room temperature state, such that any improvement of its physicals from the preliminary subzero treatment is impossible of attainment.

In carrying out the third step of my procedure, I fully plastically deform or compression reduce the steel from its subzero fixed state to both further increase or improve its physicals and to stabilize such improvement. From a practical standpoint, I prefer (if the resultant temperature of the steel after the second step is lower than room temperature) to substantially immediately effect the full plastic deformation of the steel throughout its cross section. Any subsequent work reductions or deformations may be carried out in a conventional manner without destroying the improved physicals of the steel. In this connection, I have found that the improvement in physicals is highly stable and that such improvement is not adversely affected by subsequent temperature applications in the neighborhood of 1000° F. or higher.

With reference to the composition of the steel, I find that austenite appears to be conditioned by the sub-zero temperature and fixed by the second step in such a manner that a subsequent full and uniform deformation (reduction or upset) can be effected without embrittlement. I stress the steel in the third step and attain higher physicals for a given reduction. In accordance with my invention, I attain a higher tensile level without a sacrifice of ductility, as compared to results obtained by conventional procedure. The amount of stressing is governed by the stability of the austenite content and surprisingly, may be controlled on the same basis as austenitic steels processed in a conventional manner. For example, I have found that types A, B and C alloys (as treated in accordance with steps 1 and 2 of my invention) can be deformed (reduced, drawn or upset) about as much as the same steels processed by a conventional procedure involving their direct (not subjected to steps 1 and 2 of my procedure) cold deformation from a room temperature.

The steel should initially, in its room temperature state, be at least 50% austenitic. As finally processed, it should have less than 50% ferrite and an optimum, less than 40% and above 10%. It is thus apparent that an essentially austenitic steel must be utilized which has some austenite capable of being converted to ferrite during the third step of my procedure.



Although it is difficult to determine exactly what occurs, my inventigations indicate that the sub-zero treatment produces a mixed structure in the steel, that the heat fix substantially retains such sub-zero structure (with a slight but favorable modification), so that the steel is amenable to a full development of its physicals by a full plastic or compression deformation without developing brittleness. By way of example, an austenitic steel (type B) had a tensile strength of 157,000 p. s. i. and 330 Vickers when cooled to about —75° F. in accordance with the first step of my procedure, had a tensile strength of 147,000 p. s. i. and a 310 Vickers at +2° F. after the second step was effected, and developed a tensile strength of 290,000 p. s. i. and 600 Vickers after about 70.5% deformation or reduction in accordance with the third step of my procedure. On the other hand, such a steel initially cooled to about +2° F. and tested, had a tensile strength of about 127,000 p. s. i. and a 265 Vickers.

I have also determined that less work is necessary upon an alloy processed in accordance with my invention and that the size or thickness of the alloy can be reduced to obtain physicals as good or better than those of a larger size alloy piece as processed in accordance with normal procedure. Thus, from this standpoint, an increase in ductility may be provided for a given desired tensile strength and a greater ductility can be provided in a steel shape of a required tensile strength processed in accordance with my invention, as compared to a normally processed steel of the same initial chemical composition. Another advantage of my procedure is that less deformation is required for obtaining desired physicals. For a given required tensile strength, I may employ a lesser total number of reductions, starting, of course, with an initially lesser gauge than otherwise possible. That is, lesser percentage reductions and a lesser number of passes are needed.

Austenitic steels may be processed in accordance with my invention to provide a marked improvement in physicals without breakage and without a substantial impairment of their corrosion resistance. In fact, the improvement of the physicals made possible has been sufficient to cause the usage of these more expansive steels in entirely new fields. For example, my invention makes possible, for the first time, the practical utilization of austenitic stainless wire for spring wire utilization to provide a much better spring. Heretofore, music wire which has had little corrosion resistance has been used because of its high physicals. It will be noted that the physicals obtained by my procedure run well over the minimum required for spring wire. In addition to fine wire and spring wire usefulness (a proportional yield point gain of 50% or more can be obtained in accordance with my invention), an alloy produced in accordance with my invention is particularly useful in connection with airplane requirements, since stressed parts with a minimum of weight are required.

The following Table I shows a comparison of permeability of steels A, B and C where they are processed in accordance with normal room temperature cold work procedure as compared to where they are processed in accordance with my invention (starting from the indicated, initial sub-zero temperature treatment). In this Table I, the data is based on equal percentages of deformation (reduction). It should be noted that permeability in austenitic steels is a measure of the stress to which they are subjected.

TABLE I

| Type # | H+# | Temperature | μ At 200 H |
|---|---|---|---|
| A | 9119 B | Room | 18.76 |
| A | 9119 B | —23.8° F | 35.02 |
| A | 9119 B | —58° F | 44.95 |
| B | 20315 H | Room | 2.42 |
| B | 20315 H | —23.8° F | 19.38 |
| B | 20315 H | —52.6° F | 23.02 |
| B | 20315 H | —88.6° F | 26.56 |
| C | | Room | 1.004 |
| C | | —85° F | 1.979 |

Tables II to V, inclusive, show test data on alloys A to C, inclusive, as well as on a straight chromium (non-austenitic) alloy D. The lack of improvement in alloy D is evident from Table V.

## TABLE II

*Grade A*

[C. 10 Cr 16. 97 Ni 6. 87.]

Drawn at Room Temperature to .170" Rd.
From .230" Rd., 45% Work
Ten. Size, .167" Rd.
Ult. Strength 227,000 p. s. i.
Elong. in ½", 25%
Red. of Area, 48%

| Load # | P. s. i. | Strain .001" |
|---|---|---|
| 100 | 4,550 | 2 |
| 500 | 22,750 | 10 |
| 1,000 | 45,500 | 18½ |
| 1,500 | 68,250 | 27 |
| 2,000 | 91,000 | 35 |
| 2,500 | 113,750 | 44½ |
| 3,000 | 136,500 | 54 |
| 3,500 | 159,250 | 64½ |
| 4,000 | 182,200 | 76½ |
| 4,500 | 204,750 | 90½ |
| 5,000 | 227,500 | 111 |

## TABLE II*a*

*Grade A*

[C . 10 Cr 16. 97 Ni 6. 87.]

Drawn after Subzero Treatment at −29.2° F. to .170" Rd.
From .230" Rd., 45% Work
Ten. Size, .1695" Rd.
Ult. Strength, 286,000 p. s. i.
Elong. in ½" 25%
Red. of Area, 42%

| Load # | P. s. i. | Strain .001" |
|---|---|---|
| 100 | 4,500 | 1½ |
| 500 | 22,500 | 7½ |
| 1,000 | 45,000 | 16 |
| 1,500 | 67,500 | 25 |
| 2,000 | 90,000 | 32½ |
| 2,500 | 112,500 | 41 |
| 3,000 | 135,000 | 50 |
| 3,500 | 157,500 | 59 |
| 4,000 | 180,000 | 68 |
| 4,500 | 202,500 | 77½ |
| 5,000 | 225,000 | 88½ |
| 5,500 | 247,500 | 102½ |
| 6,000 | 270,000 | 119½ |
| 6,500 | 292,500 | |

## TABLE III

*Grade B*

[C .06 Cr 18.84 Ni 8.46—Drawn at room temperature.]

| | (1) Drawn to .170" Rd. from .230" Rd., 45% Work. Ten. Size, .169" Rd. Ult. Strength, 176,000 p.s.i. Elong. in ½", 16½%. Red. of Area, 66½%. | | (2) Drawn to .145" Rd. from .230" Rd., 60.5% Work. Ten. Size, .143" Rd. Ult. Strength, 221,000 p.s.i. Elong. in ½", 15%. Red. of Area, 61%. | |
|---|---|---|---|---|
| Load # | P. s. i. | Strain .001" | P. s. i. | Strain .001" |
| 100 | 4,450 | 2 | 6,200 | 3 |
| 500 | 22,250 | 10 | 31,000 | 14 |
| 1,000 | 44,500 | 20 | 62,000 | 27 |
| 1,500 | 66,750 | 29½ | 93,000 | 40½ |
| 2,000 | 89,000 | 39½ | 124,000 | 54½ |
| 2,500 | 111,250 | 52 | 155,000 | 71 |
| 3,000 | 133,500 | 66 | 186,000 | 91 |
| 3,500 | 155,750 | 85½ | 217,000 | |
| 3,900 | 173,550 | 121 | | |



## TABLE III—Continued

*Grade B*—Continued

| | (3) Drawn to .125" Rd. from .230" Rd. 70.5% Work. Ten. Size, .124" Rd. Ult. Strength, 229,000 p. s. i. Elong. in ½", 12½%. Red. of Area, 56½%. | |
|---|---|---|
| Load # | P. s. i. | Strain .001" |
| 100 | 8,300 | 3½ |
| 500 | 41,500 | 17½ |
| 1,000 | 83,000 | 35 |
| 1,500 | 124,500 | 53 |
| 2,000 | 166,000 | 78½ |
| 2,500 | 207,500 | 97 |
| 2,800 | 232,400 | 116½ |

## TABLE III*a*

*Grade B*

[C .06 Cr 18.84 Ni 8.46—Drawn after subzero treatment at −29.2° F.]

| | (1) Drawn to .170" Rd. from .230" Rd., 45% Work. Ten. Size, .169" Rd. Ult. Strength, 193,000 p. s. i. Elong. in ½", 19% Rd. of Area, 65% | | (2) Drawn to .145" Rd. from .230" Rd., 60.5% Work. Ten. Size, .142" Rd. Ult. Strength, 243,000 p. s. i. Elong. in ½", 15½% Red. of Area, 56% | |
|---|---|---|---|---|
| Load # | P. s.i. | Strain .0001" | P. s. i. | Strain .001" |
| 100 | 4,450 | 2 | 6,300 | 3 |
| 500 | 22,250 | 10 | 31,500 | 15 |
| 1,000 | 44,500 | 19 | 63,000 | 29½ |
| 1,500 | 66,750 | 29 | 94,500 | 43½ |
| 2,000 | 89,000 | 39 | 126,000 | 58 |
| 2,500 | 111,250 | 49 | 157,500 | 75 |
| 3,000 | 133,500 | 61 | 189,000 | 94½ |
| 3,500 | 155,750 | 75 | 220,500 | 124 |
| 3,700 | 164,650 | 81½ | 233,100 | 145 |
| 4,000 | 178,000 | 93½ | | |
| 4,300 | 191,350 | 109 | | |

## TABLE III*b*

*Grade B*

[C .06 Cr 18.84 Ni 8.46—Drawn after subzero treatment at −29.2° F.]

| | (3) Drawn to .125" Rd. from .230" Rd., 70.5% Work. Ten. Size, .124" Rd. Ult. Strength, 263,000 p. s. i. Elong. in ½", 12½% Red. of Area, 48% | |
|---|---|---|
| Load # | P. s. i. | Strain .001" |
| 100 | 8,250 | 3½ |
| 500 | 41,250 | 17½ |
| 1,000 | 82,500 | 35½ |
| 1,500 | 123,750 | 52 |
| 2,000 | 165,000 | 70 |
| 2,500 | 206,250 | 91 |
| 3,000 | 247,500 | 117½ |
| 3,200 | 264,000 | 131½ |

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## TABLE IIIc

*Grade B*

[C .06 Cr 18.84 Ni 8.46—Drawn after subzero treatment at −58° F.]

| | (1) Drawn to .170″ Rd. from .230″ Rd., 45% Work Ten. Size, .166″ Rd. Ult. Strength, 222,000 p. s. i. Elong. in ½″, 22% Red. of Area, 57½% | | (2) Drawn to .145″ Rd. from .230″ Rd., 60.5% Work Ten. Siz, .143″ Rd. Ult. Strength, 254,000 p. s. i. Elong. in ½″, 11%. Red. of Area, 48% | |
|---|---|---|---|---|
| Load # | P. s. i. | Strain .001″ | P. s. i. | Strain .001″ |
| 100 | 4,600 | 2 | 6,200 | 1 |
| 500 | 23,000 | 10 | 31,000 | 12 |
| 1,000 | 46,000 | 19½ | 62,000 | 26½ |
| 1,500 | 69,000 | 29½ | 93,000 | 39½ |
| 2,000 | 92,000 | 39 | 124,000 | 53 |
| 2,500 | 115,000 | 48½ | 155,000 | 68½ |
| 3,000 | 138,000 | 58½ | 186,000 | 86 |
| 3,500 | 161,000 | 71 | 217,000 | 111 |
| 3,800 | 174,800 | 79 | 235,600 | 132½ |
| 4,000 | 184,000 | 86 | | |
| 4,500 | 207,000 | 122½ | | |

| | (3) Drawn to .125″ Rd. from .230″ Rd., 70.5% Work Ten. Size, .121″ Rd. Ult. Strength, 284,000 p. s. i. Elong. in ½″, 6% Red. of Area, 46% | |
|---|---|---|
| Load # | P. s. i. | Strain .001″ |
| 100 | 8,800 | 3½ |
| 500 | 44,000 | 16½ |
| 1,000 | 88,000 | 33½ |
| 1,500 | 132,000 | 49½ |
| 2,000 | 176,000 | 68 |
| 2,500 | 220,000 | 87 |
| 3,000 | 264,000 | 114½ |

## TABLE IIId

*Grade B*

[C .06 Cr 18.84 Ni 8.46—Drawn after subzero treatment at −90.4° F.]

| | (1) Drawn to .170″ Rd. from .230″ Rd., 45% Work Ten. Size, .169″ Rd. Ult. Strength, 232,000 p. s. i. Elong. in ½″, 25% Red. of Area, 57½% | | (2) Drawn to .145″ Rd. from .230″ Rd., 60.5% Work Ten. Size, .140″ Rd. Ult. Strength, 278,000 p. s. i. Elong. in ½″, 18½% Red. of Area, 51% | |
|---|---|---|---|---|
| Load # | P. s. i. | Strain .001″ | P. s. i. | Strain .001″ |
| 100 | 4,450 | 2 | 6,500 | 2½ |
| 500 | 22,250 | 10 | 32,500 | 12½ |
| 1,000 | 44,500 | 19 | 65,000 | 25 |
| 1,500 | 66,750 | 29 | 97,500 | 37½ |
| 2,000 | 89,000 | 37½ | 130,000 | 50 |
| 2,500 | 111,250 | 47 | 162,500 | 63 |
| 3,000 | 133,500 | 57½ | 195,000 | 77 |
| 3,500 | 155,750 | 68½ | 227,500 | 93 |
| 4,000 | 178,000 | 81 | 260,000 | 112½ |
| 4,200 | 186,900 | 86½ | 273,000 | 123½ |
| 4,500 | 200,250 | 95½ | | |
| 5,000 | 222,500 | 117 | | |

| | (3) Drawn to .125″ Rd. from .230″ Rd., 70.5% Work Ten. Size, .123″ Rd. Ult. Strength, 293,000 p. s. i. Elong. in ½″, 6% Red. of Area, 42% | |
|---|---|---|
| Load # | P. s. i. | Strain .001″ |
| 100 | 8,400 | 3½ |
| 500 | 42,000 | 17½ |
| 1,000 | 84,000 | 35 |
| 1,500 | 126,000 | 52 |
| 2,000 | 168,000 | 68½ |
| 2,500 | 210,000 | 86½ |
| 2,800 | 235,200 | 98½ |
| 3,000 | 252,000 | 106 |
| 3,400 | 285,600 | 134 |

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## TABLE IV

*Grade C*

[C .07 Ni 10.99 Cr 18.84—Drawn at room temperature.]

| | (1) Drawn to .167″ Rd. from .230″ Rd., 47.5% Work Ten. Size, .167″ Rd. Ult. Strength, 173,000 p. s. i. Elong. in ½″, 20% Red. of Area, 58% | | (2) Drawn to .145″ Rd. from .230″ Rd., 60.5% Work Ten. Size, .142″ Rd. Ult. Strength, 208,000 p. s. i. Elong. in ½″, 20% Red. of Area, 49½% | |
|---|---|---|---|---|
| Load # | P. s. i. | Strain .001″ | P. s. i. | Strain .001″ |
| 100 | 4,550 | 2 | 6,300 | 2½ |
| 500 | 22,750 | 10 | 31,500 | 12½ |
| 1,000 | 45,500 | 19½ | 63,000 | 26½ |
| 1,500 | 68,250 | 29½ | 94,500 | 41½ |
| 2,000 | 91,000 | 40 | 126,000 | 57 |
| 2,500 | 113,750 | 51½ | 157,500 | 79½ |
| 3,000 | 136,500 | 67 | 189,000 | 117½ |
| 3,500 | 159,250 | 92½ | | |
| 3,800 | 172,900 | 113 | | |

| | (3) Drawn to .125″ Rd. from .230″ Rd., 70.5% Work Ten. Size, .122″ Rd. Ult. Strength, 227,000 p. s. i. Elong. in ½″, 12½% Red. of Area, 42% | |
|---|---|---|
| Load # | P. s. i. | Strain .001″ |
| 100 | 8,600 | 3½ |
| 500 | 43,000 | 17½ |
| 1,000 | 86,000 | 35½ |
| 1,500 | 129,000 | 55 |
| 2,000 | 172,000 | 82 |
| 2,400 | 206,400 | 118½ |

## TABLE IVa

*Grade C*

[C .07 Ni 10.99 Cr 18.84—Drawn after subzero treatment at −101.3° F.]

| | (1) Drawn to .167″ Rd. from .230″ Rd., 47.5% Work Ten. Size, .166″ Rd. Ult. Strength, 197,000 p. s. i. Elong. in ½″, 20% Red. of Area, 58% | | (2) Drawn to .145″ Rd. from .230″ Rd., 60.5% Work Ten. Size, .139″ Rd. Ult. Strength, 236,000 p. s. i. Elong. in ½″, 12½% Red. of Area, 44% | |
|---|---|---|---|---|
| Load # | P. s. i. | Strain .001″ | P. s. i. | Strain .001″ |
| 100 | 4,600 | 2 | 6,600 | 3 |
| 500 | 23,000 | 10 | 33,000 | 15 |
| 1,000 | 46,000 | 20 | 66,000 | 30½ |
| 1,500 | 69,000 | 30½ | 99,000 | 44 |
| 2,000 | 92,000 | 40½ | 132,000 | 59 |
| 2,500 | 115,000 | 51 | 165,000 | 76 |
| 3,000 | 138,000 | 63½ | 198,000 | 98 |
| 3,400 | 156,400 | 75 | 224,400 | 123 |
| 3,500 | 161,000 | 78 | | |
| 4,000 | 184,000 | 101 | | |
| 4,200 | 195.200 | 120½ | | |

| | (3) Drawn to .125″ Rd. from .230″ Rd., 70.5% Work Ten. Size, .122″ Rd. Ult. Strength, 263,000 p. s. i. Elong. in ½″, 12% Red. of Area, 41% | |
|---|---|---|
| Load # | P. s. i. | Strain .001″ |
| 100 | 8,550 | 4 |
| 500 | 42,750 | 20 |
| 1,000 | 85,500 | 38½ |
| 1,500 | 128,250 | 58½ |
| 2,000 | 171,000 | 79 |
| 2,500 | 213,750 | 106 |
| 2,800 | 239,400 | 129 |

TABLE V

*Grade D*

[C .10 Cr 12.10.]

| | (1) Drawn at Room Temp. to .1895" Rd. from .244" Rd., 45% Work<br>Ten. Size, .1895" Rd.<br>Ult. Strength, 105,000 p. s. i.<br>Elong. in ½", 25%<br>Red. of Area, 65.5% | | (2) Drawn after Subzero Treatment at −101.3° F. to .1895" Rd. from .244" Rd., 45% Work<br>Ten. Size, .1895" Rd.<br>Ult. Strength, 104,500 p. s. i.<br>Elong. in ½", 25%<br>Red. of Area, 65% | |
|---|---|---|---|---|
| Load # | P. s. i. | Strain .001" | P. s. i. | Strain .001" |
| 100 | 3,550 | 2 | 3,600 | 2 |
| 500 | 17,750 | 10 | 18,000 | 10 |
| 1,000 | 35,500 | 19 | 36,000 | 18 |
| 1,500 | 53,250 | 27½ | 54,000 | 27½ |
| 2,000 | 71,000 | 36 | 72,000 | 38 |
| 2,500 | 88,750 | 49 | 90,000 | 54 |
| 2,800 | 99,400 | 65 | 100,800 | 76 |

In, for example processing wire to carry out the first step, I have placed an annealed austenitic steel coil in a large insulated tank having a removable cover, a sight glass and a coil-temperature-reading thermometer. The tank has an exit cut in it above the cooling solution level. A solution of alcohol (methyl) and Dry Ice has been used as the cooling agent or solution to produce a temperature of down to about −112° F. For temperatures down to about −320° F., liquid nitrogen may be used and a commercial freezing unit will provide a temperature of down to about −120° F. The wire may be coiled on a rotating swift or spindle in the tank and its end threaded through the exit and introduced into a contact die having a draw bore of the same diameter as the wire and a relatively large metal mass. In this die, the temperature of the wire is quickly raised to above 0° F. and the wire may then be drawn from such die into a reduction die of a smaller bore diameter at which point its physicals are fully developed and stabilized. A wiper of soft material is positioned to wipe off moisture from the wire as it leaves the tank so as to prevent the formation of frost.

In this manner, wire increments are progressively heat fixed from their subzero temperature by the first contact die and are then plastically deformed or compression-reduced in the second die to carry out the third step of my procedure. The first die has a contact bore of a conductive material such as tool steel and a sufficient mass to quickly and effectively generate heat in the wire increments passing therethrough in frictional engagement therewith. A copper coating or dry soap has been found helpful as a lubricant. Of course, it will be apparent to those skilled in the art that any suitable apparatus may be employed and that the above arrangement is merely exemplary.

This is a continuation-in-part of my application, Serial No. 241,085, filed August 9, 1951, now abandoned, and entitled "Austenitic Alloy Steel and Procedure for Making Same."

The heat fixing (highly critical second step) of my procedure involves approaching instantaneously (maximum period of less than about 5 minutes) and as quickly as possible (optimum), bringing the fully cooled austenitic steel up from its subzero temperature to a temperature above 0° F. (to a minimum of about 2 or 3° above 0° F. and as a maximum up to about 350° F.). This accomplishes several highly important results: (*a*) it is at a rate that is sufficiently fast to effect a substantial retention of the subzero-temperature-developed subzero austenitic-ferritic structure; (*b*) the steel substantially retains its unstable (subzero) austenitic condition but its (subzero) compression and tension resistance to deformation is lowered, so that it can be fully worked; (*c*) the small spread between its tensile and yield strengths which is characteristic of its subzero condition is slightly increased to an amount that is sufficient to permit fully working or plastically deforming it without developing brittleness (it has a retained favorably modified subzero structure); and (*d*) the substantial instantaneous fixing of the subzero steel makes it not only fully amenable to a full work development and stabilization of its physicals, but insures highly improved physicals by reason of the fact that it can be thereafter processed while its structure is in a retained favorably modified subzero condition.



Any suitable heating means may be employed for the heat fixing operation, such as the above-mentioned contact die for wire, or a gas or electrically-heated unit through which the sheet, strip or wire immediately passes after leaving the cooling agency or unit. The initial setting of the unit can be easily adjusted by checking the temperature of the steel (such as by a thermometer or thermo-couple), immediately as it leaves the heating unit.

In carrying out the third step of my process (which may involve one or more plastic deforming or work-reducing operations), for an optimum development of its physicals and employment of its modified subzero structure, I accomplish each work operation (from the first to the final operation) without raising the temperature of the steel above about 350° F. (maintain it substantially or well below its ageing temperature). Thus, the steel is brought down to its final gauge, shape or form after the subzero treatment and in such a manner as to develop and stabilize its highly improved physicals. It will thus be apparent that it is advantageous to effect the heat fixing step at a raised temperature that is closely above about 2° F. to provide a wider working range for the operation or operations of the subsequent plastic deforming step.

What I claim is:

1. A method of imparting improved physicals to a steel that is essentially austenitic at room temperature and that has austenite capable of being converted to ferrite under the treatment herein set forth which comprises, developing its subzero austenitic-ferritic structure by initially fully cooling the steel to a subzero temperature of below 0° F., making the steel workable and developing and retaining a favorably modified subzero structure by substantially instantaneously raising it from the subzero temperature to a temperature of at least about 2° F. and substantially below its aging temperature, and developing stabilized and highly improved physicals in the steel by then fully plastically work-stressing it throughout its section and at least initially, while retaining the steel at a temperature below about 350° F.

2. A method of imparting improved physicals to a steel that is essentially austenitic at room temperature and that has austenite capable of being converted to ferrite under the treatment herein set forth which comprises, developing its subzero austenitic-ferritic structure by initially fully cooling the steel to a subzero temperature of below 0° F., making the steel workable and developing and retaining a favorably modified subzero structure by substantially instantaneously raising it from the subzero temperature to a temperature of at least about 2° F. and up to about 350° F., and developing stabilized and highly improved physicals in the steel by then fully plastically work-deforming its favorably modified subzero structure throughout its section and at least initially, while retaining the steel at a temperature below about 350° F.

3. A method as defined in claim 2 wherein, the steel is maintained below about 350° F. while it is both initially and subsequently plastically work-deformed.

4. A method as defined in claim 2 wherein, the substantially instantaneous raising of the steel from the subzero temperature is effected within a maximum period of about five minutes.

5. A method as defined in claim 2 wherein, the steel as processed, contains not less than about 10% and not more than about 40% ferrite.

6. An austenitic steel as processed in accordance with claim 5 as characterized by its highly improved and uniform physicals throughout its section from the standpoint of ultimate strength, tensile strength, yield strength, proportional limits, and relative ductility.

7. A method of imparting improved physicals to a nickel-containing steel that contains at least 50% austenite at room temperature and that has austenite capable of being converted to ferrite under the treatment herein set forth which comprises, developing its subzero austenitic-ferritic structure by fully cooling the steel to a subzero temperature within a range of below 0° F. to −320° F.; making the steel workable and developing and retaining a favorably modified subzero structure by substantially instantaneously, and within a maximum period

of about five minutes, raising it from a subzero temperature of the defined range to a temperature of at least about 2° F. and up to about 350° F.; developing stabilized and highly improved physicals in the steel by then fully plastically reducing the steel throughout its section, while maintaining it at a temperature below about 350° F.; and effecting a maximum conversion of austenite to ferrite in the steel of 40% of its above-mentioned initial content.

8. A method of imparting improved physicals to a chromium-nickel steel that is essentially austenitic at room temperature and that has austenite capable of being converted to ferrite under the treatment herein set forth which comprises, developing its sub-zero austenitic ferritic structure by initially fully cooling the steel to a sub-zero temperature within the range of below 0° F. to —320° F., making the steel workable and developing and retaining a favorably modified sub-zero structure by substantially instantaneously and within a maximum period of about five minutes, raising it from the sub-zero temperature to a temperature of at least about 2° F. and up to about 350° F., developing stabilized and highly improved physicals in the steel by then fully plastically reducing the steel throughout its section while maintaining it at a temperature of below about 350° F. and effecting a conversion of austenite to ferrite in the steel of less than about 40% and of above about 10% of its initial room temperature content.



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