Nov. 9, 1965 E. S. NACHTMAN 3,216,868
ELEVATED TEMPERATURE WORKING AND HEAT TREATMENT OF STAINLESS STEEL
Filed March 6, 1961 6 Sheets-Sheet 1

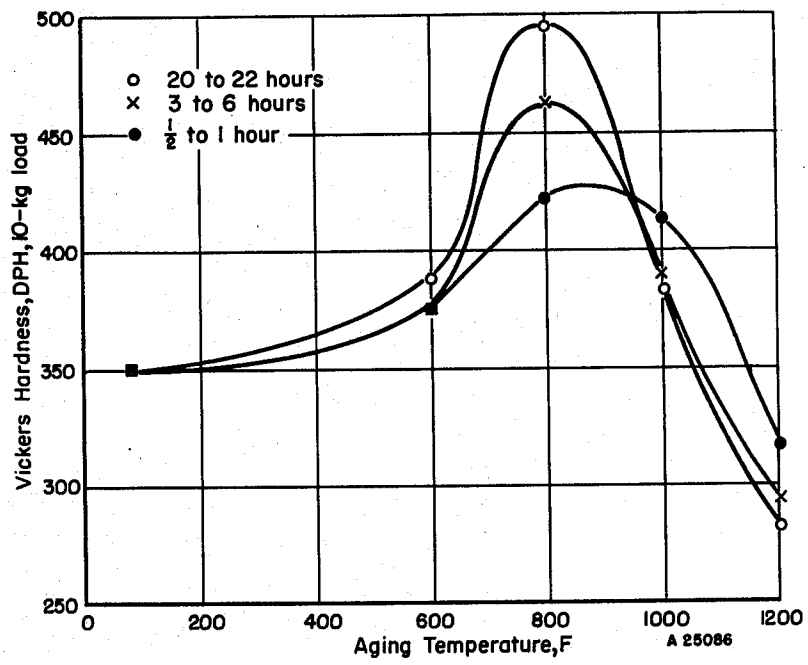


FIGURE 1 EFFECT OF AGING TEMPERATURE ON THE HARDNESS OF SOLUTION-TREATED 17-4PH STAINLESS STEEL

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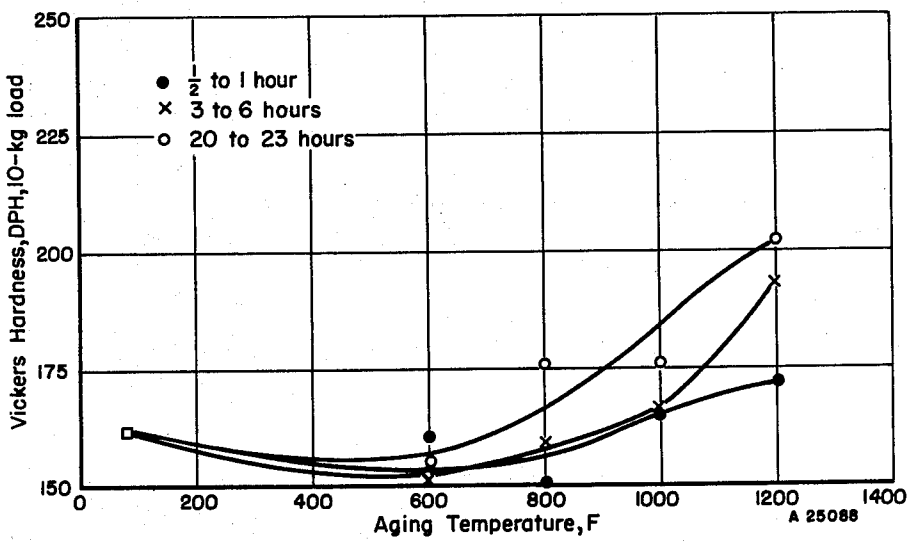


FIGURE 2 EFFECT OF AGE-HARDENING TEMPERATURE ON THE HARDNESS OF SOLUTION-TREATED 17-10P STAINLESS STEEL

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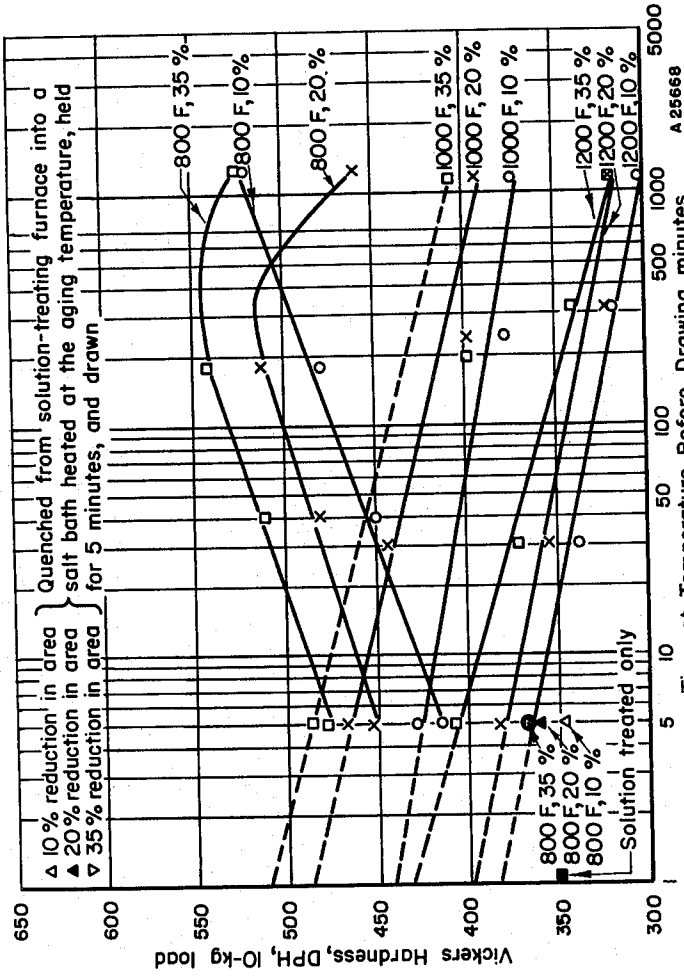


FIGURE 3 EFFECT OF TIME AT TEMPERATURE PRIOR TO ELEVATED-TEMPERATURE DRAWING OF SOLUTION-TREATED 17-4PH STAINLESS STEEL

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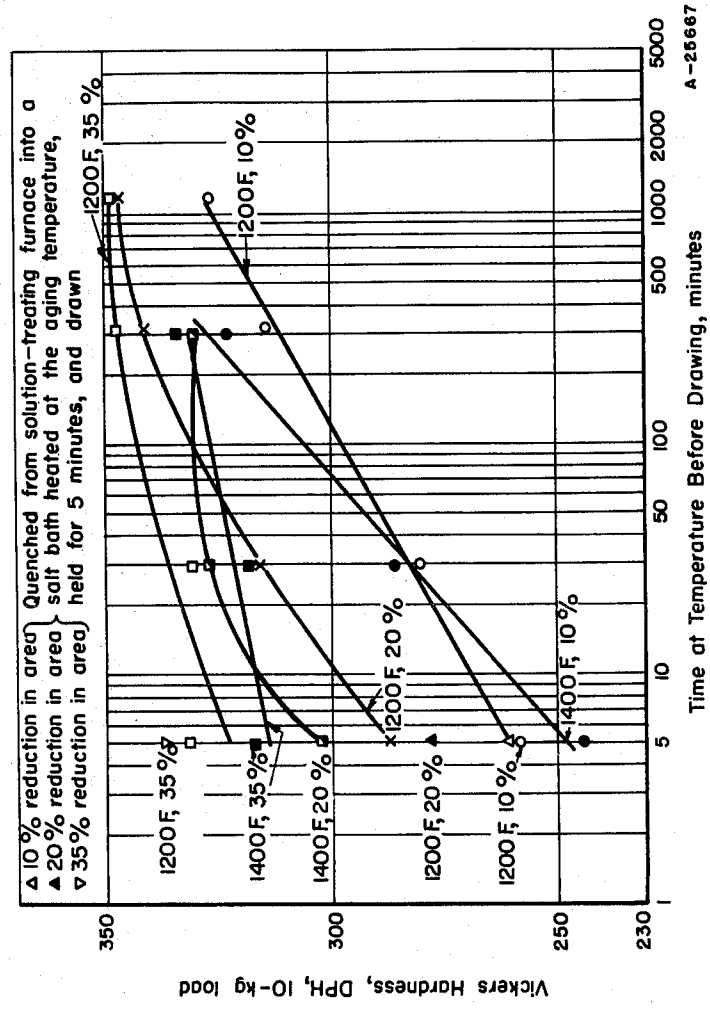


FIGURE 4 EFFECT OF TIME AT TEMPERATURE PRIOR TO ELEVATED-TEMPERATURE DRAWING OF SOLUTION-TREATED 17-10P STAINLESS STEEL

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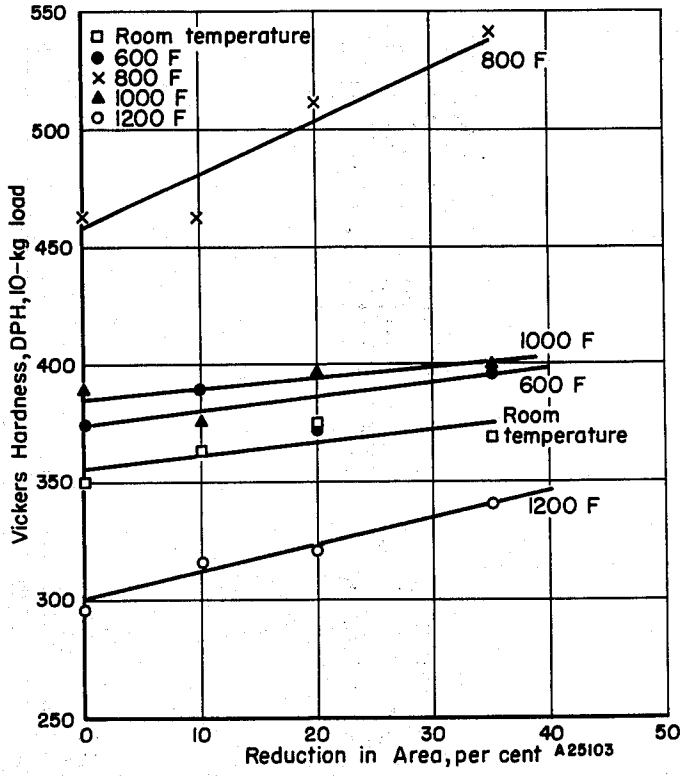


FIGURE 5 EFFECT OF DRAFT ON HARDNESS OF 17-4PH STAINLESS STEEL HEATED AT INDICATED TEMPERATURES FOR 3 TO 7 HOURS BEFORE DRAWING

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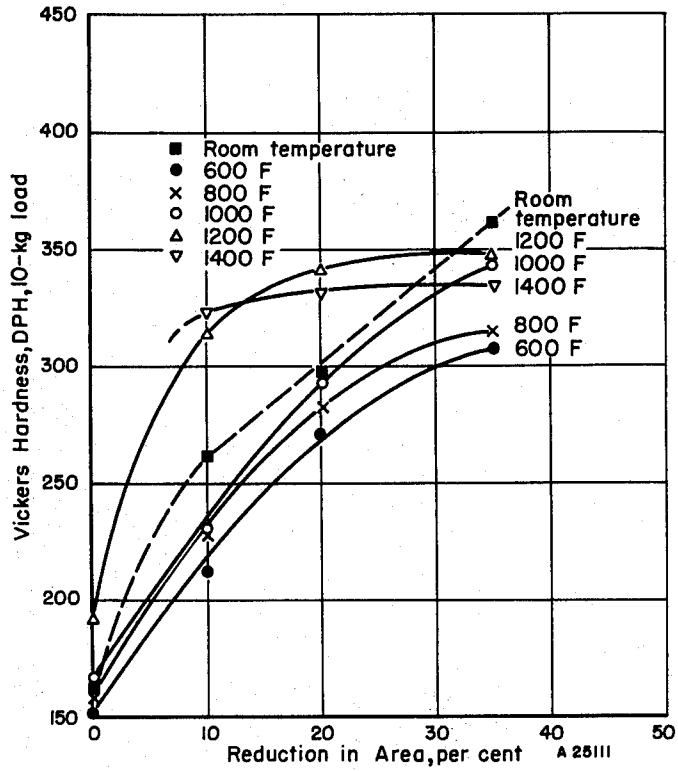


FIGURE 6 EFFECT OF DRAFT ON HARDNESS OF 17-10P STAINLESS STEEL HEATED AT INDICATED TEMPERATURES FOR 3 TO 6-1/4 HOURS BEFORE DRAWING

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United States Patent Office

3,216,868

Patented Nov. 9, 1965

3,216,868

ELEVATED TEMPERATURE WORKING AND HEAT TREATMENT OF STAINLESS STEEL

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Filed Mar. 6, 1961, Ser. No. 93,668
5 Claims. (Cl. 148—12)

This application is a continuation-in-part of my application Serial No. 74,134, filed December 6, 1960, now abandoned, for "Elevated Temperature Working and Heat Treatment of Stainless Steels."

This invention relates to a new steel having improved physical and mechanical properties, and it relates more particularly to stainless steels having these characteristics.

The products of this invention are characterized by a particularly high yield strength in combination with adequate ductility. The process is capable of producing stainless steel having the above characteristics in combination with high strength. The materials of this invention has a high yield strength to tensile strength ratio, and the process for producing such products is characterized by great commercial acceptability in that treating times may be materially reduced.

The invention has been found equally applicable to steels of the austenitic and martensitic type. The process consists broadly of working the martensitic type at temperatures below the lower critical temperature, but above 250° F., or the so-called warm working range and working the austenitic types at temperatures above about 250° F. and below the upper limit of hot working temperatures for a given austenitic steel. Temperatures in the order of about 1700° F. would be considered a practical upper limit for the austenitic varieties. The process is particularly applicable to solution-treated stainless steel of the austenitic or martensitic type which is capable of being precipitation hardened.

It is an object of this invention to provide a stainless steel having high strength, high yield strength to ultimate strength ratio, and with good ductility.

It is an additional object of this invention to provide a process for the production of stainless steel having these characteristics.

It is an additional object of this invention to provide a process which will enable the production on a commercially acceptable basis of stainless steels of the austenitic and martensitic type having these characteristics.

These and other objects of this invention will hereinafter appear and for purposes of illustration, but not of limitation, particular embodiments are shown in the accompanying drawings, in which:

FIGURES 1 and 2 illustrate the effect of aging temperature on the hardness of solution-treated stainless steel;

FIGURES 3 and 4 illustrate the effect of the time of holding at the working temperature on the hardness of stainless steel; and

FIGURES 5 and 6 illustrate the effect of the degree of working on the hardness of stainless steel.

The essential process steps of this invention include solution treating at the temperatures generally recommended for the austenitic or martensitic stainless steel being used. This solution treating is followed by cooling directly to a preselected aging or working temperature or to room temperature. Ordinarily, quenching is preferred in order to avoid undesired precipitation where this would be a possibility. The aging or working temperature will lie between the lower critical and 250° F. for the martensitic type steels and between 250° F. and about 1700° F. for the austenitic type. It has been found that an optimum aging temperature exists for each different steel. This temperature is readily obtainable by running hardness tests on specimens aged at various temperatures. The recommended aging temperature of the manufacturer will, in many cases, prove to be the temperature ideally employed in this invention. If significant aging takes place, it is followed by working at the aging temperature. However, it has been found that in many cases excellent results can be achieved if the aging is eliminated, there being only a short stabilizing period prior to the working step. In any case, the working step is preferably carried out at the temperature which is found to be the ideal aging temperature, although different temperatures within the ranges cited are contemplated.

Superior results are obtained if a longer time for precipitation is allowed prior to the working step. In this connection, it has been surprisingly found that the longer holding time prior to working, in addition to the working time itself, will be considerably less than the recommended aging time. This represents a substantial saving in heating requirements and furnace hold-up times.

Beneficial results can be obtained over a wide range of working percentages. Reductions from a few percent to 50% and above are contemplated insofar as the broad aspects of this invention are concerned.

The novel concepts of this invention may be illustrated by the following examples, wherein reference is made to the following tables.

Table 1.—Chemical compositions of two commercial precipitation-hardening stainless steels used for warm-drawing experiments

Element	Type 17-10P Heat 26146	Type 17-4PH Heat 56418
Carbon	[a] 0.12	[a] 0.041
Manganese	0.71	0.55
Phosphorus	0.27	0.021
Sulfur	[a] 0.12	[a] 0.041
Silicon	0.48	0.43
Chromium	16.51	16.99
Nickel	10.73	4.22
Copper	----------	3.58
Columbium	----------	0.33
Tantalum	----------	0.02

[a] Composition given in weight percent.

Table 2.—Room-temperature tensile properties of selected tainless steel specimens aged for short periods of time before drawing at elevated temperatures

Specimen	Stainless Steel Grade	Aging		Reduction in Area by Warm Dwg., Percent	Vickers Hardness, DPH, 10-kg. load	Yld. Str., 0.2 offset 1,000 p.s.i.	Tensile Str., 1,000 p.s.i.	Elong., Percent in ½ inch	Extension,[a] Percent	Reduction in Area, Percent
		Temp., F.	Time, Min.							
5–1	17–4PH	800	5	10	415	191	194	10	6	63
5–2						189	190	[b]	7	62
6–1	17–10P	1,200	5	10	258	76	107	68	48	80
6–2						79	108	66	48	81
7–1	17–4PH	[c] 800	5	10	348	91	158	15	10	55
7–2						91	160	[b]	10	67
8–1	17–10P	[c] 1,200	5	10	260	78	105	58	42	79
8–2						85	111	62	43	80

[a] Extension between shoulders $= \left(\frac{\text{final shoulder gage length minus original shoulder gage length}}{\text{original shoulder gage length}}\right) \times 100$.

[b] Specimen failed outside of punched gage marks.
[c] Sample quenched directly from the solution-treating furnace into the salt bath, aged for 5 minutes, and warm drawn through a die to reduce the cross-sectional area by 10 percent.

Table 3.—Room-temperature tensile properties of selected-stainless steel specimens

Specimen	Stainless Steel Grade	Aging		Reduction in Area by Warm Dwg., percent	Vickers Hardness, DPH, 10-kg. load	Yield Strength, 1,000 p.s.i.		Tensile Str., 1,000 p.s.i.	Elong., percent in ½ inch	Reduction in Area, percent
		Temp., F.	Time, Min.			0.1% Offset	0.2% Offset			
3–1	17–4PH	800	195	35	543	253	254	254	10	46
3–2						251	252	252	8	45
Aged specimen [a]	17–4PH	900	60	----------	[b] 415	----------	180	195	13	46
4–1	17–10P	1,200	315	35	350	128	132	147	40	69
4–2						131	133	147	36	68
Aged specimen [a]	17–10P	1,300	1,440	----------	[b] 286	[c] 68.5	88	137	25	39
Aged specimen [a,d]	17–10P	1,300	720	----------	[b] 302	[c] 77.5	98.5	143.5	20	32
		1,200	1,440	----------						

[a] Producer's typical properties.
[b] Brinell hardness.
[c] 0.02 percent offset.
[d] These specimens were given a double aging treatment.

Table 4.—Room-temperature hardnesses of 17–4PH and 17–10P stainless steels warm drawn after heating for short times

Grade of Stainless Steel	Drawing Temperature F.	Time at Temperature Before Drawing, Min.	Nominal Reduction of Area, Percent	Room-Temperture, Vickers Hardness DPH, 10-kg. load
17–4PH	800	5	10	415
17–4PH	800	5	20	453
17–4PH	800	5	35	479
17–4PH	1,000	5	10	428
17–4PH	1,000	5	20	467
17–4PH	1,000	5	35	486
17–4PH	1,200	5	10	368
17–4PH	1,200	5	20	384
17–4PH	1,200	5	35	408
17–4PH	[a] 800	5	10	348
17–4PH	[a] 800	5	20	363
17–4PH	[a] 800	5	35	367
17–10P	1,200	5	10	258
17–10P	1,200	5	20	287
17–10P	1,200	5	35	331
17–10P	1,400	5	10	244
17–10P	1,400	5	20	302
17–10P	1,400	5	35	317
17–10P	[b] 1,200	5	10	260
17–10P	[b] 1,200	5	20	275
17–10P	[b] 1,200	5	35	337

[a] Solution treated at 1900° F. and quenched into salt bath heated at 800° F. Rods held at 800° F. for 5 minutes and then drawn as indicated.
[b] Solution treated at 2050° F. and quenched into salt bath heated at 1200° F. Rods held at 1200° F. for 5 minutes and then drawn as indicated.

EXAMPLE 1

7/16ths inch diameter specimens made from the martensitic 17–4PH type stainless steel having the composition shown in Table 1 were solution heat-treated at 1900° F. for ½ hour and oil quenched. These specimens were then heated to about 800° F. in a salt bath and drawn through a .410 diameter die to effect a reduction of 10%. The mechanical properties resulting from this treatment are shown in Table 2, reference being made to specimens 5–1 and 5–2. The exceptionally high yield strength to tensile strength ratio, the high strength and the relatively good ductility are of particular note. The 800° F. treating temperature was selected from the data shown in FIGURE 1. As can be seen, the hardness curve obtained after the various aging temperatures provides an optimum aging temperature.

EXAMPLE 2

7/16ths inch specimens of an austenitic 17–10P stainless steel having a composition as shown in Table 1 were solution heat-treated at 2050° F. for ½ hour. The specimens were water quenched and heated to about 1200° F. in a salt bath. They were then drawn through a .410 diameter die to effect a reduction of 10%. The results are shown in Table 2, reference being made to specimens 6–1 and 6–2. It is noted that the yield strength was raised in an extremely short time. The specimens also are characterized by excellent ductility. The treating temperature of 1200° F. was selected from the curve shown in FIGURE 2. As is suggested by the curve, there is a likelihood that a higher aging time will produce a greater hardness. This is in line with manufacturers' data, which recommends temperatures above 1200° F. for optimum aging.

EXAMPLE 3

To illustrate the exceptionally high strength that can be accomplished by this process, a series of hardness tests were performed, the results of which are shown in FIGURE 3. The composition of the 17–4PH specimens was the same as that employed in Example 1. The speci-

mens having the maximum hardness were selected and tensile tests were carried out on these specimens, the results of which are shown in Table 3. The tests were performed on specimens aged at 800° F. for approximately 3–7 hours, and reduced 35% in cross-section. The mechanical testing reveals an exceptionally high yield strength to tensile strength ratio, very high ultimate strength, and adequate ductility.

EXAMPLE 4

In order to illustrate the time-saving value of the instant process while still achieving maximum hardness, specimens of the 17–10P variety having the same composition as those of Example 2 were tested. Hardness tests, the results of which are shown in FIGURE 4, reveal that a 1200° F. treating time and 35% reduction in cross-section will produce a high strength specimen. It is noted that FIGURE 4 reveals that the maximum hardness is achieved in approximately 5 hours. This is in contrast to the manufacturers' suggested aging times of 24–36 hours. The tensile tests reveal high tensile strength, high yield to ultimate ratio, and good ductility (see Table 3). It is immediately apparent that the producer's typical properties do not approach those achieved by this process.

EXAMPLE 5

In order to illustrate the effect of quenching on the properties of the austenitic and martensitic varieties, specimens were made up having the compositions of Examples 1 and 2. The 17–4PH and 17–10P specimens were treated as shown in Table 4. All the specimens were quenched to room temperature except those marked (a) and (b). The results show that with the martensitic variety, there is a substantial difference when not quenching directly to room temperature. It is equally apparent that there is substantially no effect on the specimens of the austenitic variety. Thus, with a martensitic type stainless steel, it becomes necessary to transform to the martensitic state before the working operation in order to achieve the desired properties.

The data shown in FIGURES 5 and 6 illustrates that higher strength is achieved as higher working drafts are resorted to. There is, of course, a consequent sacrifice in ductility to be expected. However, it is contemplated that drafts in the neighborhood of 50% and above could be employed.

Although the invention has been described with reference to particular steels of the austenitic and martensitic variety, the invention is intended to apply to all stainless steels, including the 300 and 400 types, and other special types such as the so-called sub-zero varieties. In the latter case, as is the case with all the martensitic types, it is necessary to transform prior to the working step. As should be obvious from the above examples, the invention is particularly advantageous when applied to steels of the precipitation-hardening type, and all stainless steels of this nature are contemplated. In this connection, the elevated temperature drawing has been shown to enhance and accelerate the aging phenomena.

It will be understood that changes may be made in the above process in a manner to provide the characteristics of this invention without departing from the spirit thereof, especially as defined in the following claims.

I claim:

1. The process of treating stainless steel hardenable by some mode of precipitation comprising the steps of solution treating the steel, cooling the steel and introducing the steel into a medium maintained at a temperature above 250° F., holding the steel at this temperature for a time sufficient to produce a substantial hardness increase in the metal without over-aging the metal, and finally working the steel while it is still maintained at this temperature to achieve a reduction of a few percent up to about 50 percent.

2. The process according to claim **1**, wherein the steel is an austenitic stainless steel and the cooling consists of quenching to room temperature.

3. The process according to claim **1**, wherein the steel is an austenitic stainless steel and the cooling consists of quenching directly into said medium.

4. The process according to claim **1**, wherein the steel is a martensitic stainless steel and the cooling consists of quenching to transform the steel to martensite.

5. The process of treating stainless steel hardenable by some mode of precipitation comprising the steps of solution-treating the steel, quenching the steel to maintain a supersaturated solid solution, reheating the steel at an elevated temperature above 250° F. and at a temperature wherein precipitation from solid solution will begin, and working the steel at elevated temperature during which precipitation will continue whereby precipitation will occur during working at the elevated temperature.

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