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J. MELILL ETAL
METHOD FOR IMPROVING THE PHYSICAL PROPERTIES OF
SEMI-AUSTENITIC STAINLESS STEELS

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2 Sheets-Sheet 1

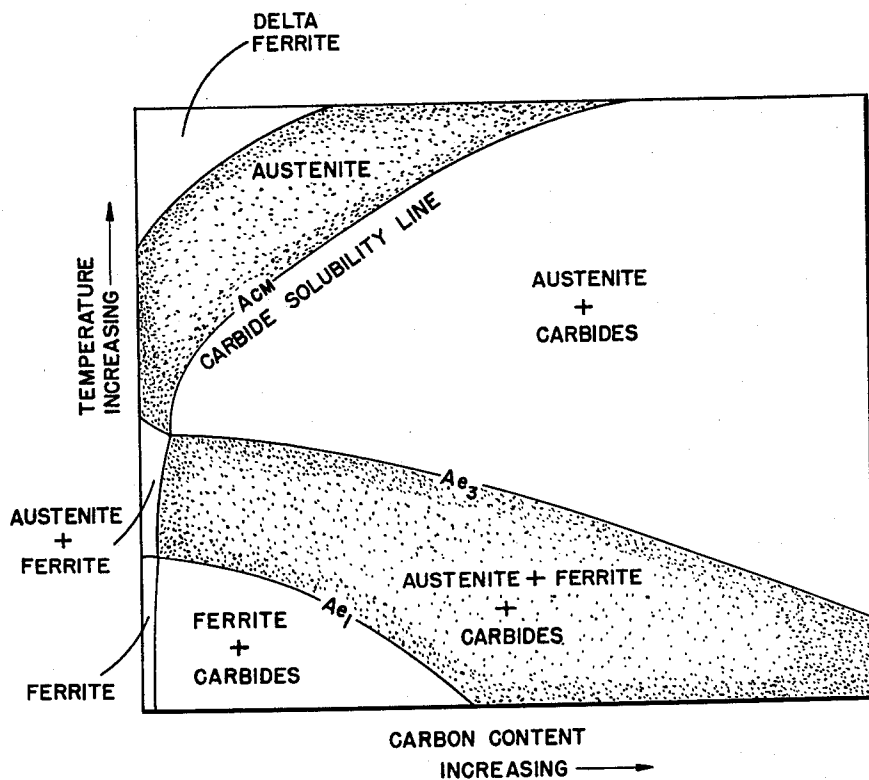


FIG. 1

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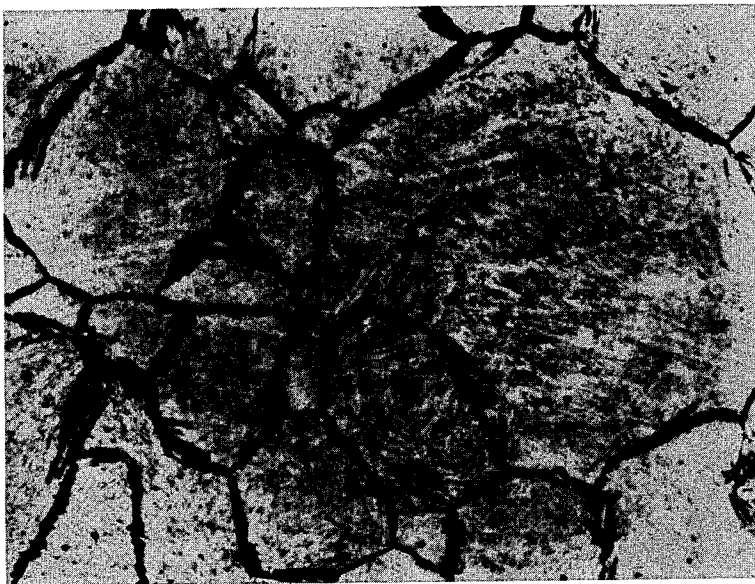


FIG. 2

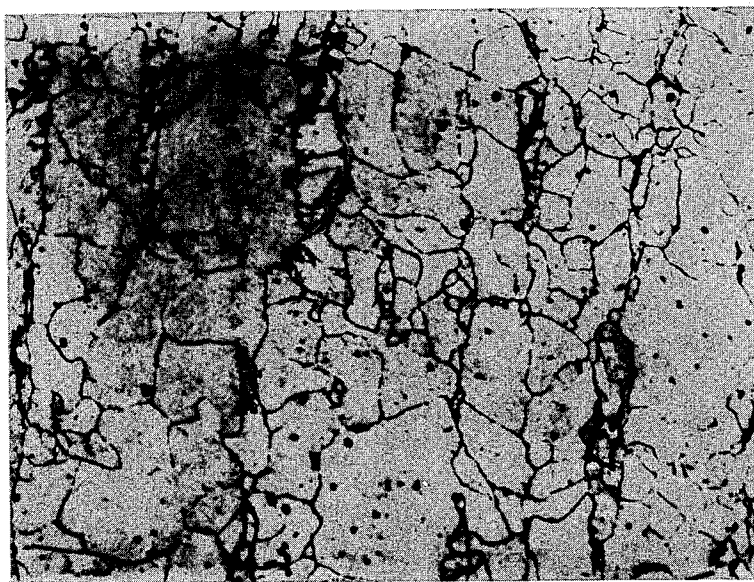


FIG. 3

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METHOD FOR IMPROVING THE PHYSICAL PROPERTIES OF SEMI-AUSTENITIC STAINLESS STEELS

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Our invention relates to the heat treatment of semi-austenitic stainless steels to improve their physical properties. It is more specifically concerned with refining the grain structure and homogenizing the alloy particles of such steels, in order to prepare them for subsequent conventional heat treatment to increase their strength and ductility.

In advanced design aircraft there is an increasing need for high-strength, corrosion-resistant alloys such as precipitation hardenable stainless steels and heat-resistant alloys. The achievement of high strength properties in these materials is quite often accomplished at the sacrifice of one or more other properties such as ductility, toughness, or impact resistance. A new class of steels, known to the art as semi-austenitic stainless steels, which are intermediate between the 300 and 400 series stainless steels in properties, have been developed for such service. The 400 series stainless steels are martensitic in structure and have a maximum of about 18 weight percent total alloy content (in the case of 431 stainless steel). The martensitic 400 series stainless steels can be converted upon heating to an austenite structure, but will revert upon cooling to room temperature again to a martensitic structure. The 300 series stainless steels generally have a minimum total alloy content of about 26 percent (for 302 stainless steel), are stable austenitic in structure at room and elevated temperatures, and are not subject to phase transformation by heat treatment. The term semi-austenitic stainless steel, as used herein and in the appended claims, is intended to designate the class of steels falling in between the 400 series martensitic and 300 series austenitic stainless steels, having a total alloy content of about 18-26 percent. These alloys resemble austenitic stainless steels in that they are soft, formable and austenitic in structure in their annealed condition. On the other hand, they resemble martensitic steels in that after hardening by refrigeration and aging, they have good strength properties and are martensitic in structure. Such steels are capable of achieving high tensile strengths, for instance over 200,000 p.s.i., and increased ductility, for instance over 10 percent elongation. The following table lists, as examples of the general class, the commercial designation and composition of some semi-austenitic stainless steels.

Alloy-----	C	Cr	Ni	Mn	Si	Mo	Al	Cb-T	Ti
AM355-----	0.12	15.5	4.50	1.00	0.40	2.75	-----	-----	-----
AM350-----	0.08	17.0	4.20	0.60	0.40	2.50	-----	-----	-----
PH15-7Mo--	0.08	15.0	7.25	0.80	-----	2.50	1.00	-----	-----
Stainless W.	0.07	17.0	7.00	0.50	0.50	-----	0.20	-----	0.70
17-7PH-----	0.09	17.0	1.00	1.00	-----	-----	1.00	-----	-----

The potentially fine properties of semi-austenitic stainless steels have not been realizable by conventional heat treatment of massive elements such as bars, billets, forgings, and extrusions constructed of such steels, as well as castings and heavy weldings. Such large pieces are used to machine out heavy fittings, and in their as-received condition from the mills are characterized by a very coarse grain structure, and display lack of homogeneity, with segregation of alloy constituents in alloy-rich and

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alloy-lean regions. In addition, upon subsequent conventional heat treatment, they are further characterized by low strength and poor ductility properties. In other words, the potentially fine properties of these materials are not achieved.

Conventional heat treatments, for instance process annealing at temperatures of about 1750° F., are resisted without improvement in physical properties. While alloy constituents are initially dissolved in the matrix at the elevated temperatures, these steels revert upon cooling to their initial condition.

It is, therefore, a principal object of our present invention to provide a conditioning treatment for improving the physical properties of precipitation hardenable, semi-austenitic stainless steels.

It is another object to condition semi-austenitic stainless steels characterized by large grains and channeling of alloy constituents into a form amenable to subsequent conventional heat treatment.

Another object is to refine the grain size and homogenize large segregated alloy constituents of semi-austenitic stainless steels.

It is a more specific purpose of our invention to provide a heat treatment conditioning method for large billets of semi-austenitic stainless steels characterized in as-received condition by large grains and segregation of alloy constituents, which refines grain structure, and provides a uniform, finely distributed network of alloy particles, and which will then permit conventional heat treatment of the conditioned steel to improve its strength and ductility characteristics.

Other objects and advantages of our invention will appear from the following description taken in conjunction with the appended claims and the attached drawings, in which:

FIG. 1 is a schematic illustration of the phase diagram representative of the semi-austenitic, precipitation hardenable stainless steels with which our invention is concerned and which generally illustrates the phase fields obtaining at various temperatures and carbon contents;

FIG. 2 is a photomicrograph ($\times 100$) of an AM355 stainless steel which has been heat treated in accordance with a conventional method; and

FIG. 3 is a photomicrograph ($\times 100$) of a section from the same piece of AM355 stainless steel which has been heat treated in accordance with our invention.

Preliminary to disclosing the details of our method, reference is made to FIG. 1. It is recognized that this diagram is representative of an equilibrium phase condition of the steel, a condition which does not normally exist. The cooling curves of the isothermal transformation diagrams of these precipitation hardenable steels are so far shifted in time as to result in metastable cooling rates when ordinary air cooling is utilized.

In any event, the various fields appearing on the FIGURE 1 equilibrium phase diagram can be defined by reference to the temperatures of demarcation between them. The A_{e1} temperature thus constitutes the demarcation temperature between a lower temperature phase which consists of ferrite and carbides and a lower intermediate temperature phase which consists of ferrite, carbides and austenite.

As is conventional, the A_{e3} temperature designates the equilibrium temperature of separation between the intermediate temperature phase field and the upper intermediate temperature phase field consisting of austenite and carbides. Similarly, the A_{cm} temperature is the equilibrium temperature between the upper intermediate temperature phase field and an upper field which consists of austenite. The A_{cm} temperature is frequently referred to as the carbide solubility line on a phase diagram since it

represents the temperature above which carbides exist in solid solution with the austenite under equilibrium conditions.

In accordance with our present invention, the physical properties of semi-austenitic stainless steels may be improved by initially heating the steel to a temperature between its A_{e1} and A_{e3} temperatures and then cooling the steel to a temperature no higher than normal room temperature. The steel is then reheated to not less than the A_{cm} temperature and is then cooled to a temperature no higher than normal room temperature. This double heat treatment, first at about the A_{e1} temperature and then above the A_{cm} temperature, greatly improves the physical properties of semi-austenitic steels initially characterized by coarse grain structure and segregated alloy constituents, and also by low strength and very low toughness. Such a steel which is initially resistant to conventional heat treatments for improving strength and ductility, can be successfully conditioned for such heat treatments by our process.

In the first step of our process, the semi-austenitic stainless steel is heated to a temperature between its A_{e1} and A_{e3} temperatures in order to unstabilize the composition by expelling alloy constituents from solution, particularly chromium and carbon, which are austenite stabilizers. The steel is heated in this step to a relatively modest temperature between about the A_{e1} and A_{e3} , and preferably at or just above A_{e1} , in order to cause the precipitation reaction, but yet low enough to unstabilize the material so that it converts upon cooling to a form other than the original austenite form. The temperature within the A_{e1} - A_{e3} will vary depending upon the nature of the steel, for instance with greater total alloy content the A_{e1} and A_{e3} are lowered. However, in general we find that a temperature of about 1300-1550° F. is very satisfactory for the first heat-treating step, while about 1375° F. is optimum. Heating above the indicated temperature results in a lower driving force to the reaction, making it, even if feasible, impractically long. The length of the heat treatment will vary with temperatures employed and the nature of the material. While the time the steel is retained at the indicated temperature is not critical, we find that at least about two hours is generally very satisfactory, and about three hours is optimum.

The steel is then cooled to a temperature no greater than about normal room temperature, generally by air cooling. Upon cooling, the steel goes through a phase transformation to the lower ferrite form. The term ferrite is used herein generically to embrace such structures as martensite, bainite and pearlite. While air cooling accomplishes substantially complete phase transformation, the steel may be sub-zero cooled, say to about -100° F., to insure complete phase transformation.

The first heating and cooling step with the phase transformation from austenite to ferrite significantly refines the grain structure. However, the alloy constituents are not homogenized and large segregates are still found at the grain boundaries. Therefore, the ferrite steel is reheated to a temperature above the carbide solubility line, A_{cm} , in order to redissolve the precipitated alloy phases in the metal matrix to produce a completely homogeneous fine-grained structure. The reheating step results in the substantial transformation of the matrix to austenite. The reheating cycle, thus, essentially transforms the matrix to austenite, causes uniform dissolution of the precipitated alloy constituents into the matrix, and results in a refined, homogeneous grain structure.

As indicated before, the temperature corresponding to A_{cm} phase region is dependent upon the nature of the alloy. We find, though, that a temperature of about 1750-2000° F. is generally very satisfactory for the reheating step and that a temperature of about 1850° F. is optimum. Although still higher temperatures will serve to redissolve and homogenize the precipitated alloy con-

stituents, grain growth will also occur, and hence temperatures higher than indicated are not desirable. The time requirements for the second heating step are somewhat shorter than for the first. The time at temperature should be at least about one-half hour, and about one hour is optimum.

The steel, which now has its alloy constituents redissolved in a uniform manner in a fine grain structure, may be cooled to a temperature no higher than approximately room temperature.

The foregoing two-cycle heating and cooling process converts the semi-austenitic stainless steel into a form which is now amenable to normal heat treatments such as tempering or process annealing, for improving the strength and ductility of the steel. For example, the steel may be subject to a process anneal at a temperature of about 1750° F., followed by quenching, sub-zero cooling, and aging of the final martensite structure.

The following examples are offered to illustrate our invention in greater detail:

Example 1

An AM355 steel forging was heated to 1375° F. and retained at that temperature for a period of three hours, after which it was cooled to room temperature. The forging was then sub-zero cooled at -100° F. for three hours. It was then reheated to a temperature of 1850° F., retained for a period of one hour and cooled to room temperature. The forging was then subsequently heat treated in the conventional manner by reheating to 1750° F., cooled to a temperature of -100° F. in order to insure completion of the martensite transformation and finally reheated to a temperature of 850° F. and held at that temperature for two hours in order to accomplish tempering.

A comparative steel forging was treated in accordance with a conventional method by heating to a temperature of 1750° F. for a period of one hour, cooling to a temperature of -100° F., and subsequently tempering the martensite by reheating to a temperature of 850° F. and retaining at that temperature for a period of two hours.

The physical properties of the conventionally treated AM355 forging and the forging treated in accordance with our method are indicated by the photomicrographs of FIGURES 2 and 3 which, respectively, illustrate the micro-structure of the comparative specimens.

Reference to FIGURE 2 will readily demonstrate that the grain size of the conventionally treated AM355 forging is greatly in excess of standard ASTM grain size 1, while the grain size of the specimen treated in accordance with our method, as shown in FIGURE 3, has a mean size range of approximately 5. It is also important to note that the conventionally heat-treated AM355 steel has a very heavy, continuous network of brittle grain boundary precipitate, while the steel treated in accordance with our method has a very fine and substantially discontinuous grain boundary. It will be clear from a visual inspection of FIGURES 2 and 3 that the ductility of the conventionally treated steel is substantially less than that of the steel treated by our method.

These conclusions are substantiated by destructive tensile tests performed on coupons selected from each of these specimens, as indicated in the following table:

Physical Properties	Coupon of Fig. 2 (Average of three coupons)	Coupon of Fig. 3 (Average of three coupons)
Ultimate Tensile Strength.....p.s.i.	216,000	216,000
Yield Strength.....p.s.i.	169,000	177,000
Elongation in 1".....percent.	3.5	12

It will be seen that the heat-treatment method of our invention not only substantially increased the yield strength, while maintaining the ultimate tensile strength

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of one precipitation hardenable stainless steel, but, more importantly, increased elongation by a factor of more than three. Since it is generally considered that the minimum elongation requirement of a high tensile strength stainless steel should be well above 3½ percent, the conditioning method described herein will permit the alteration of precipitation hardenable stainless steels to materials of construction having high strength, ductility, toughness and resistance to impact failure. An elongation increase of such magnitude, while maintaining tensile strength and increasing yield strength, is clearly demonstrative of the improved result to be obtained by practicing our invention.

It is to be understood that the foregoing description is by way of illustration only and not by way of limitation; the accompanying claims setting forth the limits of our invention.

We claim:

1. A method of conditioning semi-austenitic, precipitation hardenable stainless steel which comprises heating said steel to a temperature between about the A_{e1} - A_{e3} temperatures to precipitate alloying constituents from the matrix, cooling to a temperature no higher than normal room temperature to effect phase transformation to ferrite, thereby resulting in grain refinement with alloying constituents concentrated along grain boundaries, reheating said steel to not less than the A_{cm} temperature, to dissolve the alloying constituents within the austenitic matrix, and thereafter cooling to a temperature no higher than normal room temperature to form a fine grain structure with homogeneous distribution of alloying constituents in the matrix.

2. The method of claim 1 wherein the steel is retained at the A_{e1} - A_{e3} temperature for a period of at least about two hours, and at not less than the A_{cm} temperature for a period of at least about one-half hour.

3. A method of conditioning semi-austenitic precipitation hardenable stainless steel originally characterized by large grain structure and segregation of alloying constituents, which comprises heating said steel to about the A_{e1} temperature to cause precipitation of alloying constituents from the matrix and unstabilize the structure, cooling to a temperature no higher than the normal room temperature to transform austenite to ferrite, thereby refining grain structure, reheating to not less than the A_{cm} temperature to cause dissolution of alloy constituents distributed along grain boundaries within the resulting austenite matrix, and thereafter cooling to a temperature no higher than the normal room temperature, thereby producing a fine grain ferrite structure having homogeneous distribution of alloying constituents within the matrix.

4. A method of conditioning semi-austenitic precipitation hardenable stainless steel initially characterized by a large grain structure and non-uniform distribution of

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alloying constituents, which comprises heating said steel to a temperature of about 1300-1550° F. to cause precipitation of alloying elements from solution, cooling to a temperature no higher than about the normal room temperature to effect phase transformation to ferrite, thereby refining the grain structure, reheating to a temperature of about 1750-2000° F. to dissolve the alloying constituents distributed along the grain boundaries within the austenite matrix, and thereafter cooling to a temperature no higher than the normal room temperature, thereby obtaining ferrite of fine grain structure with homogeneous distribution of alloying constituents within the ferrite matrix.

5. The method of claim 4 wherein the first heat treatment is conducted for a period of at least about two hours, and the second heat treatment is conducted for a period of at least about one-half hour.

6. A method of conditioning a semi-austenitic precipitation hardenable stainless steel having a total alloy content of about 24 weight percent and initially characterized by large grain sizes and non-homogeneous distribution of alloy constituents, which comprises heating said steel to a temperature of about 1375° F. to cause precipitation of alloying constituents from the matrix and the formation of austenite, thereafter air cooling the steel to a temperature no higher than normal room temperature, to produce a ferrite structure of fine grain size with alloying constituents concentrated along grain boundaries, reheating the steel to a temperature of about 1800° F. to cause dissolution of alloying constituents within the austenite structure, and then air cooling the steel to a temperature no higher than normal room temperature to produce a ferrite structure of fine grain sizes with homogeneous distribution of alloying constituents within the ferrite matrix.

7. The method of claim 6 wherein the steel is heated at the first-named temperature for a period of about three hours, and at the second-named temperature for a period of about one hour.

8. The method of claim 6 wherein the alloying constituents of the steel comprise, by weight percent, approximately: 0.12 C; 15.5 Cr; 4.50 Ni; 1.00 Mn; 0.40 Si; and 2.75 Mo.

9. The method of claim 7 wherein the steel is sub-zero cooled in the first cooling step.

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