Characterization of Precipitation-Hardenable Stainless Steels

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Abstract: - Precipitation hardening (PH) stainless steels are used where high strength and good corrosion resistance are required as well as for applications requiring high fatigue strength, and stress corrosion resistance. The present work consists of comparison of the conventional cooled PH steels with the cryogenic cooled PH steels. Hardness, proof stress, ultimate tensile strength properties were characterized for the above steels. Metallographic studies were carried out to observe the changes in the microstructures of the above mentioned steels. The study revealed that the PH steels that are conventionally cooled have less hardness than the cryogenic cooled samples. The same trend was also observed incase of mechanical properties. The microstructures showed some amount of refinement in case of cryogenic treated samples due to lower temperatures involved which was responsible for complete transformation of austenite to martensite.

Key words : PH steels, Precipitation hardening, Cryogenic cooling, Hardness, Microstructure

I. INTRODUCTION:

A. PH steels : Precipitation hardening stainless steels use nickel and chromium as their major alloying elements and are a combination of the martensitic and austenitic alloy types. In the solution annealed condition these steels have a semi-austenitic structure allowing for good machining and forming characteristics. However this is transformed after machining to a martensitic structure during heat treatment and strength is increased as a result. One of the advantages of precipitation hardening stainless steels is that full heat treatment can be achieved at relatively low temperatures minimizing distortion.

Precipitation hardening stainless steels are used where high strength and good corrosion resistance are required as well as for applications requiring high fatigue strength, good resistance to galling and stress corrosion resistance. The reduced distortion during heat treatment makes these steels particularly suitable for intricate parts requiring machining and welding and where freedom from distortion is a requirement. Precipitation hardening stainless steels are used in aerospace, defense and offshore oil & gas industries. For missile components, motor shafts, valve stems, gears and other mechanical components.

The high tensile strengths of precipitation hardening stainless steels come after a heat treatment process that leads to precipitation hardening of a martensitic or austenitic matrix. Hardening is achieved through the addition of one or more of the elements Copper, Aluminium, Titanium, Niobium, and Molybdenum.

B. 13-8 PH Steel:

13-8 VIM-VAR steel is a precipitation hardening martensitic stainless steel offering excellent fracture toughness and transverse mechanical properties coupled with the resistance to stress-corrosion cracking and high strength characteristics common to the family of precipitation hardening steels. A wide range of mechanical properties can be realized by selecting various single cycle low temperature aging treatments. The alloy is double vacuum melted (VIM-VAR…vacuum induction melted followed by vacuum arc re-melting) to consistently assure low gas content, improved homogeneity and superior cleanliness.

Table 1: Chemical composition of 13-8 PH steel

Element	**C**	**P**	**Si**	**Ni**	**Cr**	**Al**
Composition (wt%) max	0.05	0.01	0.1	7.5 to 8.5	12.3 to 13.3	0.9 to 1.35
Element	**Mn**	**S**	**Mo**	**N**	**Fe**	
Composition (wt%) max	0.1	0.008	2-3	0.01	~74	

C. 15-5 PH steel:

15-5 PH stainless steel offers high strength and hardness with excellent corrosion resistance. SS 15-5 can be age-hardened by a low temperature treatment. 15-5 PH stainless steel offers good corrosion resistance and high strength therefore has been used for aerospace applications.

Table2: Chemical composition of 15-5 PH steel

Element	**C**	**P**	**Si**	**Ni**	**Cr**
Composition (wt%) max	0.07	0.04	1.0	3.5 to 5.5	14.0 to 15.50
Element	**Mn**	**S**	**Cb+ Ta**	**Cu**	**Fe**
Composition (wt%) max	1.0	0.30	0.15 to 0.45	2.5 to 4.5	~72

D. Cryogenic Cooling:

Cryogenics is the branch of physics which deals with the production and effects of low temperatures. Low temperatures means that the temperatures that are produced are far less than 0˚C. The effects of such low temperatures are very different from the ordinary temperatures. The cryogenic cooling indicates cooling with the coolant which has the temperature less than zero such are liquid N_2, liquid CO_2 and liquid CH_4. These coolants produce intense cold temperatures which are of order -195.8˚C, -78.4˚C and -265˚C temperatures respectively. Some applications of cryogenics are Magnetic resonance imaging (MRI), Electric power transmission in big cities, Frozen food, Forward looking infrared (FLIR), and Blood banking etc.

E. Cryogenics in METALLURGY:

In metallurgy, cooling is a common process where five types of cooling are generally employed, namely furnace cooling, air cooling, oil, brine and water quenching. Several stresses are included in these metals and then these are relieved by annealing of these metals at a temperature less than the hardening temperature so that the properties doesn't change and internal stresses are being relieved. Treating those metals in similar manner some properties will not change and there will be improvement in some properties such as wear resistance, tensile and fracture properties. Cryogenic cooling is newly implemented in the metallurgy where the stress relieving process is done at lower temperatures by cooling with a coolant which is in solid or liquid form, which melting or boiling point is less than -15˚C . Generally employed coolants are liquid nitrogen or liquid carbon-di-oxide or liquid methane. The main importance of this cooling is cooling a material below sub-zero temperature so as to relieve stresses and to improve wear resistance. The conventional cooling is different from the cryogenic cooling is that it uses ice as coolant where the minimum temperature reached is 5˚C. When we consider alloy steels which has high alloying content generally the martensitic transformation gets below zero degrees centigrade. So the complete transformation of martensite is not possible and retained austenite phase in the steels decreases its hardness. To complete obtaining of martensite the steel has to be cooled below 0˚C for the complete transformation. This is done by the cryogenic cooling if the material. The basic principle involved in these studies is maintaining a continuous phase in the steel and only one phase. Our study includes cryogenic cooling of PH steels. This also includes comparison of properties obtained by the cryogenic cooling to those obtained by the conventional cooling. Not all the ph steels are employed but to our study we take a sample of 15-5 PH steel, 13-8 PH steel. Main properties of these samples are being taken into consideration for comparison and these include hardness, micro structure, impact testing, ultimate tensile stress and proof stress. The composition of those steels and applications are listed in details.

II. EXPERIMENTAL WORK:

A. Cryogenic cooling of 15-5 PH and 13-8 PH steels:

Fig.1 shows the steps involved in precipitation hardening and cryogenic hardening. The precipitation hardening of the materials takes place in two steps whereas cryogenic tempering takes place in three steps. The steel samples used were H1050 (13-8 PH) and H1025 (15-5 PH). **Fig. 2** shows solution treatment for 13-8 PH steel and 15-5 PH steel respectively where the process consists of heating the samples to a certain temperature and giving sufficient soaking time followed by air cooling of the samples.

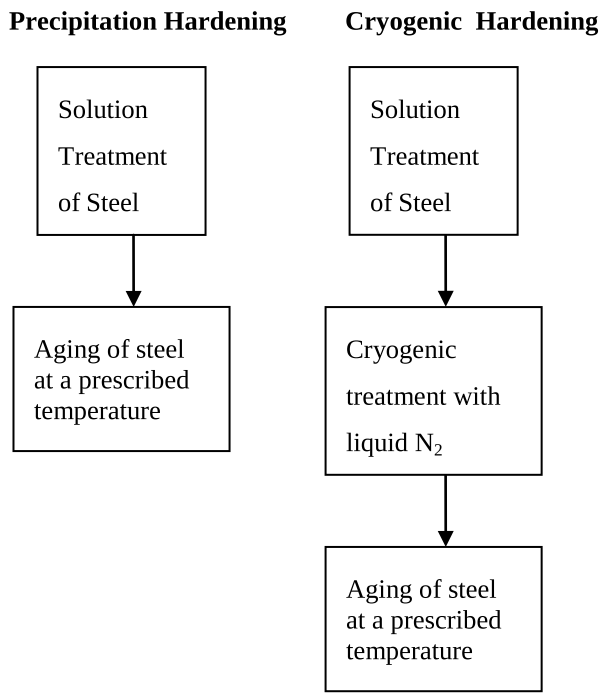


Fig 1: The difference in the cycles of precipitation hardening and cryogenic hardening

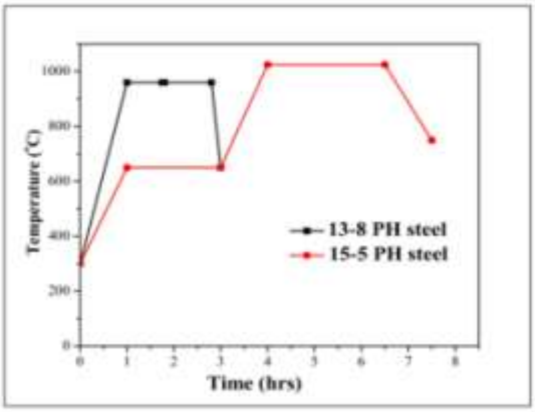


Figure 2: Solution treatment for 13-8 and 15-5 PH steels

B. Process of cryogenic treatment:

Fig. 3 shows the cryogenic heat treatment cycle for both the samples in general. The steps involved are (a) After analyzing the size the size of material the holding time is being calculated for the solution treatment. (b) The solution treatment of the steel is carried out at the prescribed temperature depending on the recrystallization temperature and (c) Then the cooling is done in air. Then the steel sample is transferred to the cryogenic chamber where the cryogenic treatment is carried out.

Then in cryogenic chamber the following heat treatment cycle is being employed (a) The sample is being warmed in air to room temperature and (b) The sample is then aged at the prescribed temperature and then again air cooled. The various conditions of the aging temperatures are defined and the temperature selection depends on the final properties to be obtained.

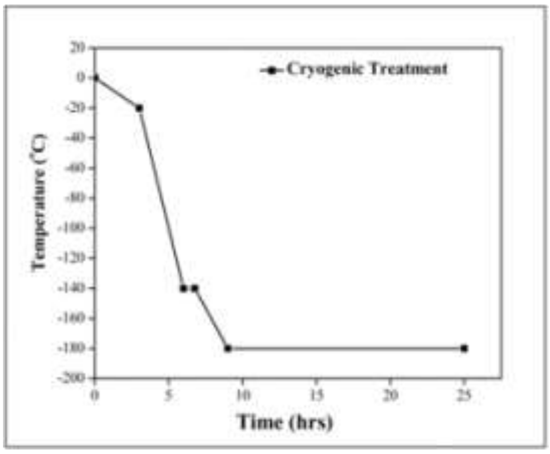


Figure 3: Cryogenic heat treatment cycle

III. RESULTS & DISCUSSION :

A. Hardness:

The **Brinell** scale characterizes the indentation hardness of materials through the scale of penetration of an indenter, loaded on a material test-piece. The typical test uses a 10 mm (0.39 in) diameter steel ball as an indenter with a 3,000 kgf (29 KN; 6,600 lbf) force. For softer materials, a smaller force is used; for harder materials, a tungsten carbide ball is substituted for the steel ball. The indentation is measured and hardness calculated as:

$$\mathrm{BHN} = \frac{2P}{\pi D\left(D - \sqrt{D^2 - d^2}\right)} \quad \text{Eq. (1)}$$

Where P = applied force

D = diameter of indenter

d = diameter of indentation

Table 1: Comparison of hardness values

Type of PH steel	Condition	Hardness in conventional process	Hardness in cryogenic Cooling
13-8	H1050	385 BHN	477 BHN
15-5	H1025	375 BHN	435 BHN

Table 1 shows the hardness values of the steels which are hardened by conventional precipitation hardening method and also hardened by cryogenic treatment. It can be seen that the steels which are conventionally cooled have less hardness than the cryogenic cooled samples. This is due to complete transformation of austenite to martensite and the presence of intermetallics. In cryogenically treated samples 13-8 PH steel exhibits a higher hardness value due to more number of intermetallic phases present (Ni_3Al and NiAl).

B. Proof Stress and Ultimate Tensile Strength:

The proof stress for 13-8 and 15-5 PH steels is given in Table 2 and Table 3 shows the ultimate tensile strength for 13-8 and 15-5 PH steels .The proof stress and the ultimate tensile strength of the cryogenically treated samples exhibited higher values. This is due to the complete precipitation of the intermetallics and alloy carbides in case of cryogenically treated samples.

Table 2: Comparison of proof stress for 13-8 and 15-5 PH steels

Proof Stress				
Conventional Cooling			Cryogenic Cooling	
Type of PH steel	Load (KN)	Proof Stress (MPa)	Load (KN)	Proof Stress (MPa)
13-8	35.44	1040	41.05	1337
15-5	26.35	864	34.77	1144

Table 3: Comparison of and ultimate tensile strength for 13-8 and 15-5 PH steels

Ultimate Tensile Strength (UTS)				
Conventional Cooling			Cryogenic Cooling	
Type of PH steel	Load (KN)	UTS (MPa)	Load (KN)	UTS (MPa)
13-8	36.49	1077	42.06	1370
15-5	28.40	932	35.69	1175

C. Microstructure:

Fig. 4 (a) and (b) show the microstructures of 13-8 PH steel in conventional precipitation hardened process and in cryogenic treatment respectively. Similar microstructures are shown in case of 15-5 PH steel in **Fig. 5** (a) and (b). It can be seen that there is some amount of refinement in the microstructures of cryogenically treated samples. This is due to low temperatures involved in case of cryogenically treated samples where sufficient time is given for complete transformation of austenite to martensite.

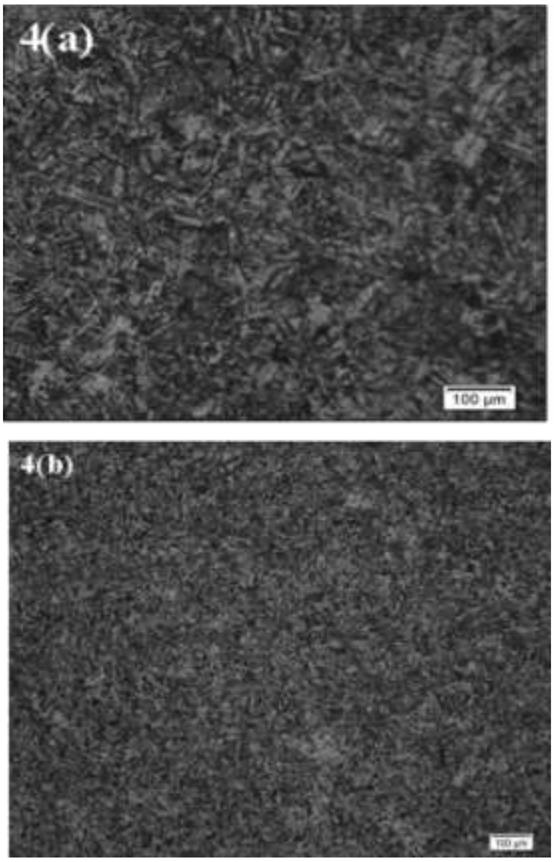
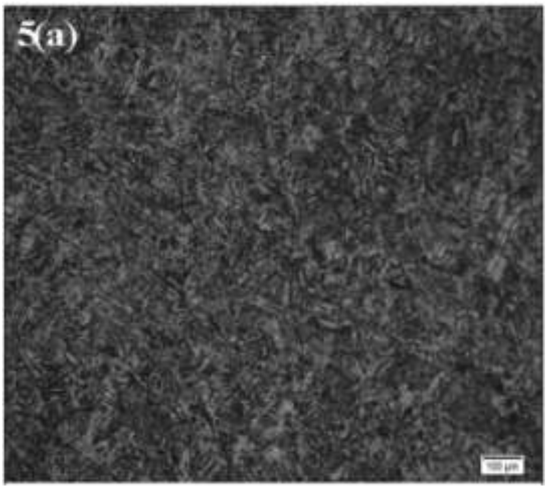


Figure 4 : Optical microstructures of 13-8 PH steel (a) Conventional Cooling (b) Cryogenic Cooling



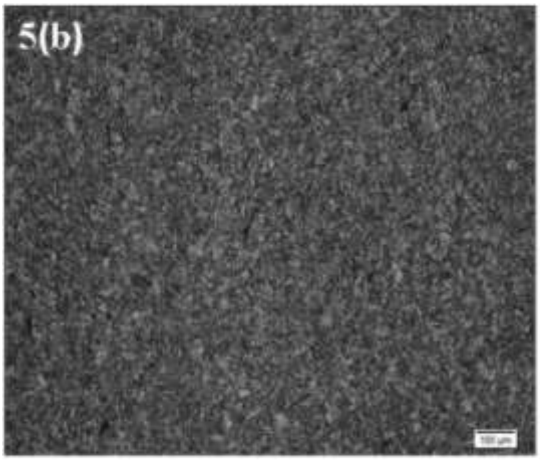


Figure 5 : Optical microstructures of 15-5 PH steel (a) Conventional Cooling (b) Cryogenic Cooling

IV. CONCLUSIONS:

The steels that are conventionally cooled have less hardness than the cryogenic cooled samples. This is due to complete transformation of austenite to martensite. The tensile properties are also improved incase of cryogenic treated samples/ The cooling with liquid N_2 is found to improve the desired properties satisfactorily. The dimensional accuracy of the sample is maintained. The microstructures reveal some amount of refinement in case of cryogenic treated samples

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