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Effect of Aging on Mechanical Properties of S465 Stainless Steel

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Abstract: Applications requiring high strength stainless steels are growing rapidly. Precipitation hardened stainless steels have been limited usage in powder metallurgy despite their high strength. It is possible to induce strengthening and precipitation hardening in austenitic and martensitic stainless steels by adding suitable alloying elements such as copper, niobium which forms intermetallic precipitates during aging. The present work is focused on influence of titanium on mechanical behavior of precipitation hardened stainless steels. The precipitation hardened stainless are basic material for the high strength applications. Heat treatment processes such as solution annealing followed by aging were conducted to enhance mechanical properties like hardness and tensile properties. The solution annealing was carried out at 950°C for all samples. Aging was carried out at different temperatures from 500°C-600°C and soaked for regular intervals of time. Mechanical properties were observed by Micro hardness testing equipment and Universal testing machine (UTM). Then the samples were examined by Optical microscope and Scanning electron microscope. It is observed that during peak aging period the size of the precipitates are enough to hold the dislocation tightly. Thus hardness and strength are increased.

Keywords: Precipitation Hardening Stainless Steel; Aging, Heat treatment, Titanium, Micro hardness, Tensile Properties.

I. INTRODUCTION

The challenge with most engineering materials is finding something that is soft and enough to be formed into a useful shape and then strong enough to be particular use. This use is reflected in the spring material that is in use today. Steel wire or strip can be hardened primarily in two ways: cold working, or quenching and tempering. Stainless steels can also be hardenable in a similar fashion, but an additional strength path exists called “age hardening” or “precipitation hardening” [1]. In 1906, precipitation hardening stainless steels was discovered on the aluminum-copper alloy called “Duralumin” by the German metallurgist Alfred Wilm. It took about 15 years after this finding to fully understand and then exploit the mechanism of precipitation hardening. This discovery provided aluminum an extra level of strengthening that enable it alloys to be used in the high-strength applications. The growth of modern aircraft industry would not have been possible without this development. Many high-strength alloys have been using this mechanism, which is not only evident in aluminium but also in cobalt, nickel, copper, and titanium alloys [1]. Stainless steels are defined as iron based alloys containing at least 10.5 % chromium and a maximum of 1.2% carbon. Stainless steels may contain nickel as another major alloying element, with a content of up to 38 % plus other alloying elements and stabilizers. The chromium content renders stainless steels corrosion resistant [2, 3]. There are more than hundred grades of stainless steel. Stainless steels are classified into five groups: Austenitic, martensitic, Ferritic, Duplex and Precipitation hardening [2]. The four of these classes are defined based on the metallurgical crystalline microstructure of the material. Precipitation hardening stainless steel, as the fifth class is based on the material’s ability to be hardened by conventional heat treatment. Precipitation hardening stainless steels are alloys of iron-chromium-nickel with the addition of one or more precipitation hardening elements such as aluminium, titanium, copper, niobium and molybdenum. These steels are designed to be formable in the solution annealed condition and can be subsequently be hardened by treating to strength levels. The driving force for the development of Precipitation Hardening Stainless Steels is to develop corrosion resistant steels with strength and toughness levels superior to martensitic stainless steels. By suitable alloying additions and appropriate heat treatment, it is possible to induce precipitation hardening in austenitic and martensitic stainless steels.

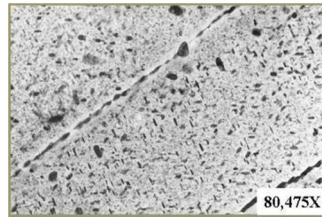
Today these steels are available in a wide range of products – bars, wires, plates, sheets [4]. Precipitation hardening stainless steels are developed as materials for Air craft fittings, Missiles, Nuclear waste casks, Turbine blades, Mechanical components, Instance retaining rings, Jet engine parts, Oil, gas and nuclear industries, Rotors of centrifugal compressors, Springs, chains, valves, Gears,

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Aircraft parts and Pressure vessels, fasteners.

II. LITERATURE SURVEY

Precipitation hardening stainless steels are iron-nickel-chromium alloys containing precipitation hardening elements such as aluminum, titanium, copper, niobium and molybdenum. Precipitation hardening (PH) stainless steels derive strength due to the precipitation of fine coherent precipitates [6]. The major advantage when compared to martensitic stainless steels is that the PH steels can be fabricated in annealed condition and subsequently precipitation hardened at lower temperatures. Such treatment eliminates the need for fast cooling and attendant problems like warpage and cracking due to martensitic transformation. The strength and hardness of some metal alloys may be enhanced by the formation of extremely small uniformly dispersed particles of a second phase within the original phase matrix. This is accomplished by appropriate heat treatments. The process is called **precipitation hardening** because the small particles of the new phase are termed "**precipitates**". [5]



"Age hardening" is also used to designate this procedure because the strength develops with time, Alloys that are hardened by precipitation treatments include Al-Cu, Cu-Be, Cu-Sn, and Mg-Al; and some ferrous alloys. The composition of the precipitation hardening steels is dictated by three factors viz., the electron-atom ratio, the atomic size and the compressibility.

PH steels can be made to austenitic, semi austenitic and martensitic types. PH stainless steels have high strength, good toughness and corrosion resistance both at ambient and elevated temperatures. PH stainless steels find extensive use in aircraft structures, landing gears, armament and other engineering applications. The steels achieve high strength by the precipitation of Ni_3Al , NiAl or copper in a martensitic or austenitic matrix.

A. Types of PH Stainless Steels

Precipitation hardening stainless steels are classified according to microstructure obtained after solution heat treatment viz., martensitic, semi-austenitic and austenitic [7, 8].

B. Austenitic

Austenitic precipitation hardening steels retain their austenitic structure after annealing and hardening by ageing. At the annealing temperature of 1095 to 1120°C the precipitation hardening phase is soluble. It remains in solution during rapid cooling. When reheated to 650 to 760°C, precipitation occurs. This increases the hardness and strength of the material. Hardness remains lower than that for martensitic or semi-austenitic precipitation hardening steels austenitic alloys remain nonmagnetic. In austenitic class, the matrix is a solid solution of iron containing transition elements like manganese, cobalt and nickel.

C. Semi-Austenitic Alloys

Semi-austenitic PH stainless steels were developed for increased formability before the hardening treatment. These alloys are completely austenite in the quenched condition after solution annealing (which displays good toughness and ductility in the cold-forming operations), and eventually martensite can be obtained by conditioning treatment or thermo-mechanical treatment. Ultrahigh strength can be obtained in these steels by combinations of cold working and aging.

D. Martensitic

Martensitic precipitation hardening stainless steels have a predominantly austenitic structure at annealing temperatures of around 1040 to 1065°C. Upon cooling to room temperature, they undergo a transformation that changes the austenite to martensite. In Fe-Cr-Ni martensite, many substitutional elements produce precipitation hardening. The effect of these elements on precipitation hardening varies significantly. While Ti and Cr have strong influence on Al, Nb, Mo and V have moderate effect and Cu, Co and Zr have less effect. Many high strength precipitation hardening martensitic steels are so alloyed that the strengthening is derived

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primarily from the precipitation of coherent ordered precipitates such as NiAl as in 13-8 grade or BCC copper rich precipitates or FCC copper precipitates as in 15-5 and 17-4 grades. Depending on the ageing temperature and time, the size and sometimes the shape of the precipitates vary.

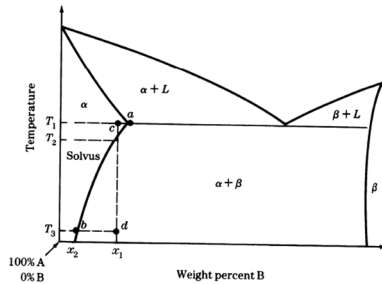


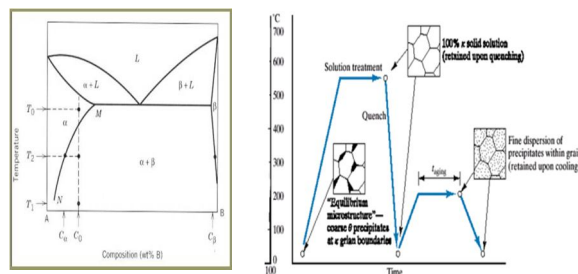
Fig. 1. Binary phase diagram for two metals A and B having a terminal solid solution which has a decreasing solid solubility of B in A with decreasing temperature.

E. Strengthening Process Of Precipitation Hardening Steels [9, 10]

1) *Solution Heat Treatment:* The alloy is heated to a relatively high temperature that allows any precipitates or alloying elements to dissolve completely. During this process all solute atoms are dissolved to form a single-phase solid solution. The solution heat treatment generally takes place in 950 to 1066°C [9].

2) *Quenching:* After solution heat treatment, the alloy is rapidly quenched to create a super saturated solution. The quenching can be done in air, water and oil media. The cooling rate must be sufficiently rapid to create a super saturated solution. The faster the cooling rate the finer grain size is formed which leads to improve mechanical properties.

3) *Aging:* The supersaturated solid solution is heated to an intermediate temperature within the two-phase region. At this temperature diffusion rates become appreciable. The precipitates of the second phase form as finely dispersed particles.



III. SCOPE OF PRESENT WORK

To study the influence of precipitates in mechanical properties by various heat treatments in PH stainless steel.

The detailed study consists of following steps

To carry out solutionising at $950 \pm 5^\circ\text{C}$, to dissolve all the precipitates present in the alloy and followed by air cooling to form super saturated solution.

To carry out aging heat treatment 510°C , 530°C , 550°C , 570°C at 2 hours, 5 hours, 10 hours, 24 hours and 48 hours.

To evaluate Micro and Marco hardness for base metal and aged samples.

To carry out the microstructural characterisation for base metal and for under aged, peak aged and overaged samples.

To carry out the tensile test for under aged, peak aged and overaged samples at different strain conditions at room temperature.

IV. EXPERIMENTAL PROCEDURE

The material for present study has been receiving from melting chamber was done in Furnace. The ingots were hot forged into plates by using hydraulic press further the plates were hot worked to the desired sizes - Then the test material were cut down into

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required dimensions i.e., 10mmx10mmx12mm samples by using CNC Wire cut E.D.M Wire cut machine- After sectioning, all samples are subjected to different heat treatment conditions prior to testing. The heat treatment was done under open atmosphere, without causing any incipient melting. Solution and Aging treatment is carried out in air furnace. The furnaces used for these heat treatment is Muffle Furnace - Solution treatment is given to S465 alloy to strengthen it by refining the γ' precipitate size. Thus produced microstructures exhibit maximum strength - 510°C - 570°C with interval of 20°C and soaked for 2 hr, 5 hr, 10 hr, 24 hr and 48 hrs. Strengthening as a result of precipitation hardening takes place - (OM)The samples to be examined for optical microscopy were first polished using different grades of emery paper grades like 180,400,600,800 and 1000 (SiC papers). After preliminary polishing, the samples were final polished on the cloth with various grades like 9 μ , 3 μ , 1 μ and 0.5 μ using diamond pastes. The samples were then thoroughly cleaned with acetone. The cleaned samples were subsequently etched using an etchant consisting of copper chloride 1 gm., hydrochloric acid 25ml, nitric acid 10ml and water 50 ml. After etching samples were observed under metallurgical microscope at 100X and 200X magnifications and the resulted microstructures are shown in Results - All the samples were thoroughly cleaned using ultrasonic container, half filled with acetone. Samples were observed under secondary electron (SEM) Mode at different magnifications by using EDAX Quanta 400 Scanning electron microscope. The resulted SEM microstructures are shown in Results - Macro hardness: Rockwell hardness tester is used to measure the micro hardness for all samples. This test measures the depth of penetration instead of the diameter of the indentation - Micro hardness: It measures all the samples by using the Vickers's Micro hardness Tester with a square base diamond pyramid indenter with an included angle of 360^o between the faces were used during hardness measurement. The resulted hardness values are shown in Results - Tensile tests were conducted by using INSTRON 5500R universal testing machine. The samples were tested for each heat treatment and the values of the tensile properties were reported.

V. RESULTS & DISCUSSIONS

A. From Hardness Measurement

Micro-hardness and Macro-hardness on the samples assessed in all conditions in Rockwell and Vickers scales and the hardness measured across the surface of the sample and also the average hardness recorded. As seen from the readings it is observed that sample in condition II - 510°C/2hrs has maximum hardness.

B. From Optical Microscope

Microstructures examined under optical microscope at 200X and 500x in etched condition. As seen from the samples in etched conditions (Fig. 4.2 (a) to 4.2 (b)) it is observed that the carbides aligned around the grain boundaries with discontinuous network, occasionally in intra-granular sites. In condition II the particle are fine and discontinuous.

CONDITION-I (original condition)

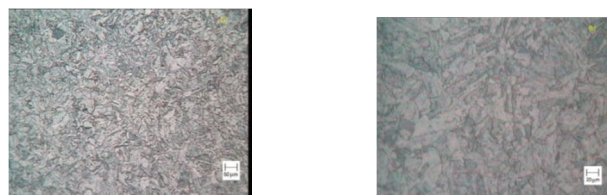


Fig. 4.2 (a) Micrographs of the condition I of the S465 alloy in (a,b) etched conditions, with magnification of 200X and 500X.

CONDITION-II (530-2 hrs)

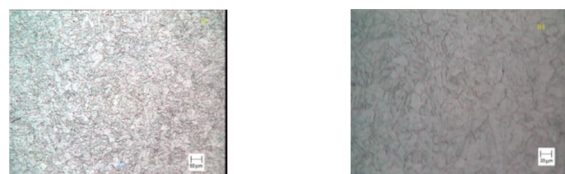


Fig. 4.2 (b) Micrographs of the condition II of the S465 alloy in (a,b) etched conditions, with magnification of 200X and 500X.

C. Scanning Electron Microscope

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The etched samples were examined under scanning electron microscope for finer Microstructural features. During the aging period, spherical inter metallic compounds and fine carbides precipitated. The PH stainless steel sample aged at 530°C-2 hours the microstructure contains martensite laths within the grain and also it contains fine sized precipitates within the matrix and it rises the strength of an alloy. The precipitates present in this condition are mainly occurs due to presence of titanium and it decreases movement of dislocation. The figure 4.3 (a) & (b) shows the microstructure of aging samples.

Tensile Specimen	0.2 % YS (MPa)	UTS (MPa)	% Elongation	% Reduction in area
Condition I	809	931	18	17
Condition II	1379	1445	17	12
Condition III	1360	1414	18	13
Condition IV	1348	1386	18	12

CONDITION-I (As received condition)

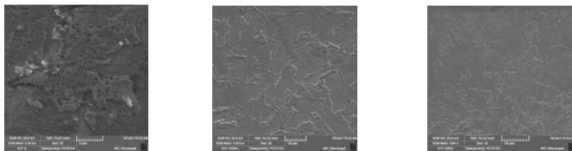


Fig. 4.3 (a) Micrographs of the condition I of the S 465 A alloy in etched conditions, at magnifications of 5000X, 1000X, 500X (SE)

CONDITION-II (530-2 hrs)

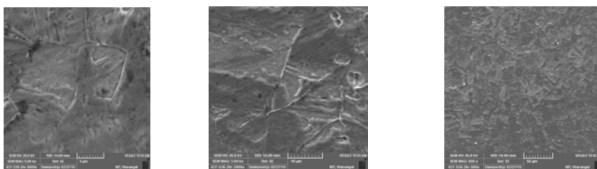


Fig. 4.3 (c) Micrographs of the condition II of the S 465 A alloy in etched conditions, at magnifications of 5000X, 1000X, 500X (SE)

D. Tensile Properties

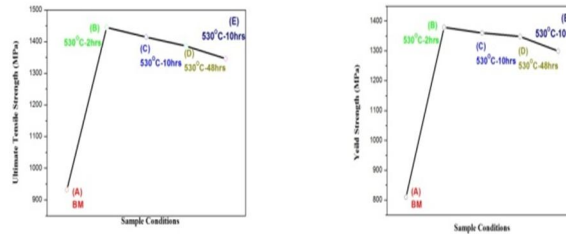
From the hardness profile it is observed that 530°C aging has uniform profile. So the tensile test is carried out at room temperature in different condition is given below.

Condition	Temperature
Condition I	Base Sample
Condition II	530 ⁰ C/2 hrs
Condition III	530 ⁰ C/10 hrs
Condition IV	530 ⁰ C/48 hrs

As seen from the test results it is observed that sample tested from condition II has resulted in higher yield strength and tensile strength and the sample tested from heat treatment condition III slightly inferior in properties to that of condition II. The sample

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tested in as received condition has given the lowest yield strength and tensile strength. During optimum aging the precipitates hold the dislocations tightly results in high strength. The following table shows the properties obtained on the samples tested in different conditions. Table: Tensile properties of S465 in as received and our different heat treatment conditions.



VI. CONCLUSION

The present work carried out effect of aging on mechanical properties of S465 stainless steel and it also deals with the scope of future work on the same material.

The effect of titanium on hardness was observed in all conditions of heat treatment and tensile behavior and microstructural characterization were observed in five conditions.

Better hardness and tensile strength were observed at 530⁰ C aged sample soaked for 2 hrs. At this condition the precipitates stop dislocation movement and hold them tightly, thus hardness and strength are increased.

From microstructures it is observed that during over aging the precipitates are randomly distributed and distance between precipitates is high thus results in lower strength.

Due to addition of titanium in PH stainless steel results in high strength to weight ratio particularly developed for landing gear components, aerospace applications (like in Missiles), springs, Chains, Values, Gears, Pressure vessels and Oil/Gas/Nuclear industries.

Precipitation hardened stainless steel, as class; offer the designer a unique combination of fabricability, strength, easy of heat treatment and corrosion resistance not found in any class of materials.

VII. SCOPE FOR FUTURE WORK

Development and Manufacturing of several grades of materials viz., Austenitic, ferritic, Martensitic and precipitation hardened grade, with good combination of mechanical and elevated temperature properties have been the main focus of many aeronautical industries for the last two decades.

High strength to weight ratio considerations have been the prime considerations for achieving higher efficiencies of aero engines. As a result many advanced special steels and super alloys with different combinations of chemistry and heat treatments to modify the microstructure combinations to enhance high temperature strength have been emerged.

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