**International Journal of Engineering Research and Development**
*e-ISSN: 2278-067X, p-ISSN: 2278-800X, www.ijerd.com*
*Volume 9, Issue 1 (November 2013), PP. 22-26*

# Effect of Precipitation Hardening on Microstructural Characteristics of 15-5 Ph Steel

R.Sri Rama Devi[1], J.Sai nath[2], Ms. C.Usha Kiranmai[3]
[1]*(Associate Professor), Department of Metallurgical Engineering, JNTUHCEH, Kukatpally-500028.*
[2]*(Senior lecturer), Department of Metallurgical Engineering,JN.Govt Polytechnic-Hyd*
[3]*(Lecturer), Department of Metallurgical Engineering, JNTUHCEH, Kukatpally-500028.*

**Abstract:-** 15-5 PH (Fe-Cr-Ni) is a precipitation hardening, martensitic stainless steel. The alloy has good strength, transverse toughness and ductility, hardness capability and corrosion resistance compared with 304 stainless. It may be used in the solution treated condition or heat treated to obtain a wide variety of properties.15-5 PH stainless steel is used in applications requiring high strength and toughness in all directions. It is mainly used in aircraft components and actuator parts for modern fighter aircrafts. The main aim of this project is to study the effect of precipitation hardening on micro structural characteristics of 15-5 PH stainless steel. A round specimen of 15-5 PH with 10mm diameter & 100mm length is selected. Chemical analysis is done using optical emission spectrometer. Specimen is cut into small specimens with dimensions 10mm diameter & 10mm thickness using abrasive cutting technique. Precipitation hardening is done for each sample at different temperatures as per AMS 2759 standards. Hardness of solution annealed and precipitation hardened samples is determined by using Rockwell hardness tester. Microstructures were observed by using Metallurgical microscope under the magnification of 100X.

## I. INTRODUCTION

15-5 PH is a delta ferrite-free compositional modification of 17-4 PH precipitation hardening martensitic stainless steel containing less chromium and slightly higher nickel. Vacuum arc re-melting lowers the alloys gas content, reduces and disperses inclusions, and minimizes alloy segregation during solidification. These factors, coupled with the elimination of delta ferrite, result in superior transverse toughness compared to 17-4 PH.

Precipitation hardenable martensitic stainless steel 15–5 PH (Fe–15Cr–5Ni–4Cu) exhibits excellent combination of high strength and hardness, with excellent corrosion resistance, weld ability, forgeable and finally amenability to cast and machine. Additional features of this alloy also include high resistance to crack propagation, good transverse properties and good resistance to stress-corrosion cracking in marine atmosphere. The 15–5PH alloy exhibits good short transverse ductility and a reasonably high strength in large section sizes. This steel is normally used in either annealed or over-aged condition and is normally heat treated after fabrication. However, caution should be exercised as the parts of this steel should never be used in heat treatment condition ‘A’.

Two different heat treatments involving precipitates can alter the strength of a material: solution heat treating and precipitation heat treating. Solid solution strengthening involves formation of a single-phase solid solution via quenching. Precipitation heat treating involves the addition of impurity particles to increase a material's strength. Precipitation hardening via precipitation heat treatment is the main topic of discussion in this project.

## II. EXPERIMENT

The main aim of the project is to study the effect of precipitation hardening on micro structural characteristics of 15-5 PH stainless steel.

### CHEMICAL ANALYSIS

A round specimen of 15-5 PH stainless steel is selected with 10mm diameter & 100mm length. Chemical analysis is done using optical emission spectrometer. Using abrasive cutting specimen is cut into small specimens 10mm diameter & 10mm thickness.

### SOLUTION TREATMENT (Condition ‘A’)

15-5 PH stainless steel is a martensitic precipitation hardening stainless steel. The material supplied from the mill is in solution annealed condition. Solution annealing involves formation of a single-phase solid solution via quenching.

1038 ± 14°C/soaking time 30 min + cool below 32°C/OQ or AC.

**PRECIPITATION HEAT TREATMENT**

Precipitation hardening is also known as age hardening. Precipitation hardening is done to the each sample at different temperatures as per the AMS 2759 standard.

| condition | temperature (°C) | temperature (°F) | soaking time | cooling media |
|---|---|---|---|---|
| H900 | 482°C | 900°F | 1 Hour | Air cool |
| H925 | 496°C | 925°F | 4 Hours | Air cool |
| H1025 | 552°C | 1025°F | 4 Hours | Air cool |
| H1075 | 580°C | 1075°F | 4 Hours | Air cool |
| H1100 | 593°C | 1100°F | 4 Hours | Air cool |
| H1150 | 621°C | 1150°F | 4 Hours | Air cool |

**HARDNESS TESTING**

Hardness testing is done for all samples using Rockwell hardness 'C' scale Diamond indenter with maximum load of 150 kg.

**METALLOGRAPHY SAMPLE PREPARATION**

Mounting is carried on Automated Mounting Machine. All the samples are mounted in thermosetting plastic (Bakelite) using one inch mount. All the mounted samples are polished and etched for microstructure evaluation.

**ETCHING**

Etching means subjecting the surface of the metal to chemical attack. Etching is done for all samples to reveal the microstructure. The etching reagent used is modified fry's reagent.

Composition of the Modified fry's reagent is

Water $H_2o$ - 100ml.

Hydrochloric acid HCl - 50ml.

Nitric acid $HNO_3$ - 25ml.

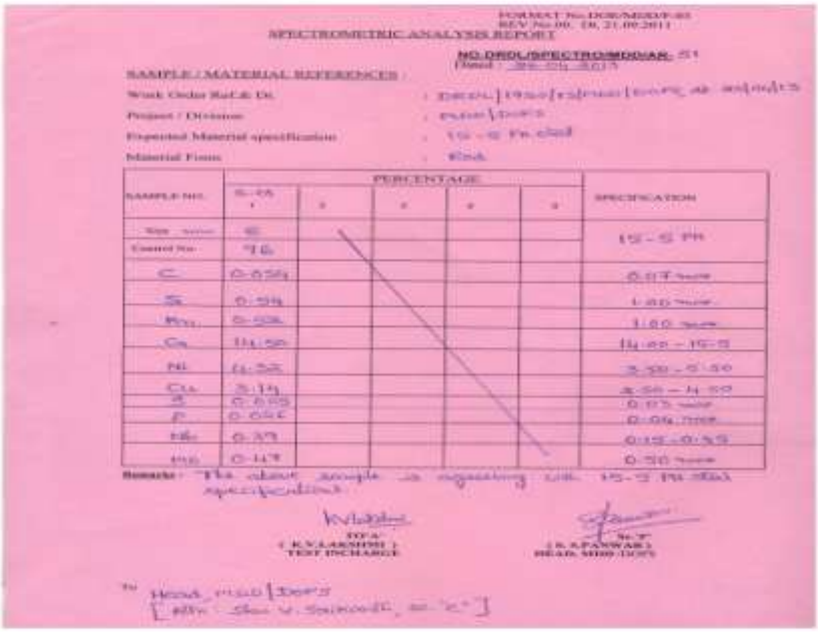
Copper chloride $CuCl_2$ - 1gm.

**MICROSTRUCTURE EVALUATION**

After etching all the samples are ready for microstructure examination. Microstructures are observed using optical microscope.

## III. RESULTS

Spectrometric analysis report


SPECTROMETRIC ANALYSIS REPORT

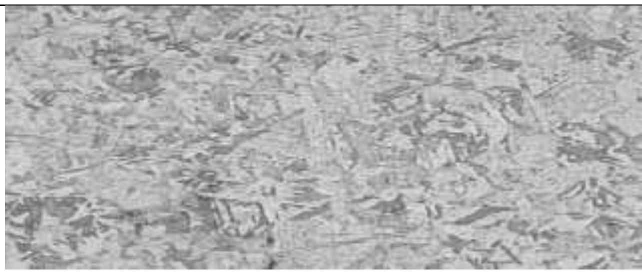
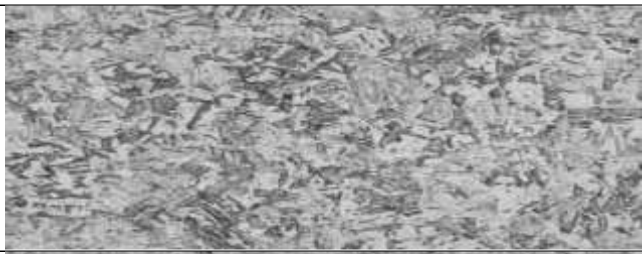
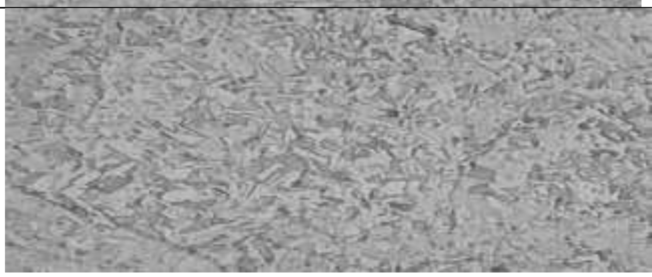
SAMPLE / MATERIAL REFERENCES :

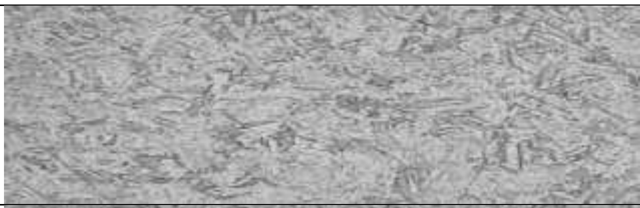
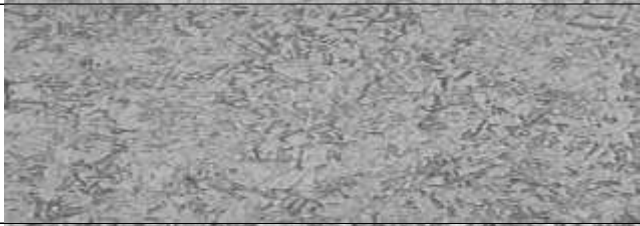
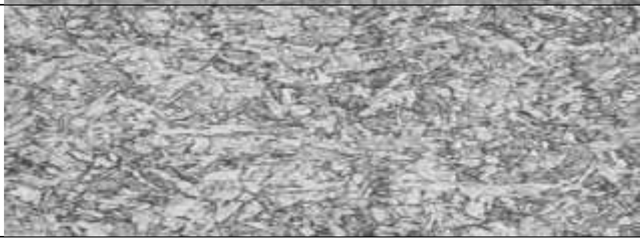
| SAMPLE NO. | PERCENTAGE | | | | | SPECIFICATION |
|---|---|---|---|---|---|---|
| | 1 | | | | | |
| | 76 | | | | | 15-5 PH |
| C | 0.056 | | | | | 0.07 max |
| Si | 0.54 | | | | | 1.00 max |
| Mn | 0.58 | | | | | 1.00 max |
| Cr | 14.50 | | | | | 14.00 - 15.5 |
| Ni | 4.52 | | | | | 3.50 - 5.50 |
| Cu | 3.14 | | | | | 2.50 - 4.50 |
| S | [illegible] | | | | | 0.03 max |
| P | [illegible] | | | | | 0.04 max |
| Nb | 0.37 | | | | | 0.15 - 0.45 |
| Mo | [illegible] | | | | | 0.50 max |

**HARDNESS VALUES**

| **Condition** | **Hardness in HRC** |
|---|---|
| Solution annealed | 36 HRC |
| H900 condition | 38 HRC |
| H925 condition | 38 HRC |
| H1025 condition | 39 HRC |
| H1075 condition | 37 HRC |
| H1100 condition | 33 HRC |
| H1150 condition | 35 HRC |

**MICROSTRUCTURES OF 15-5 PH STEEL**

| | |
|---|---|
| **1. Condition:** Solution annealed **Orientation:** Circumferential **Microstructure:** Martensite **Etchant:** Modified fry's reagent **Magnification: 100X.** |  |
| **2. Condition:** Solution annealed **Orientation:** Longitudinal **Microstructure:** Martensite **Etchant:** Modified fry's reagent |  |
| **3. Magnification: 100X. Condition:** H900 **Microstructure:** Martensite **Etchant:** Modified fry's reagent **Magnification: 100X.** |  |
| **4. Condition:** H925 **Microstructure:** Martensite **Etchant:** Modified fry's reagent **Magnification: 100X.** |  |
| **5. Condition:** H1025 **Microstructure:** Martensite **Etchant:** Modified fry's reagent **Magnification: 100X.** **Condition:** H1075 |  |

| | |
|---|---|
| **6. Microstructure:** Martensite **Etchant:** Modified fry's reagent **Magnification: 100X.** |  |
| **7. Condition:** H1100 **Microstructure:** Martensite **Etchant:** Modified fry's reagent **Magnification: 100X.** |  |
| **8. Condition:** H1150 **Microstructure:** Martensite **Etchant:** Modified fry's reagent **Magnification: 100X.** |  |

## IV. SUMMARY AND DISCUSSION

The main aim of the project is to study the effect of precipitation hardening on micro structural characteristics of 15-5 PH stainless steel.15–5PH (Fe–15Cr–5Ni–4Cu) is Martensitic Precipitation hardening stainless steel. The material supplied from the mill is in annealed condition.

Chemical analysis is done by using optical emission spectrometer, Cr, Ni, Cu, Nb, Mo are determined as the basic alloying elements. Chemical analysis test results have been enclosed here with for verification.

Using abrasive cutting eight samples have cut from bar specimen, out of them six samples are subjected to precipitation hardening treatment at different temperatures as per the AMS2759 standard. And other two samples were retained in solution annealed condition for micro structural evaluation.

The hardness values obtained by Rockwell hardness testing are enclosed here with for verification.

| **Condition** | **Hardness in HRC** |
|---|---|
| Solution annealed | 36 HRC |
| H900 condition | 38 HRC |
| H925 condition | 38 HRC |
| H1025 condition | 39 HRC |
| H1075 condition | 37 HRC |
| H1100 condition | 33 HRC |
| H1150 condition | 35 HRC |

Precipitation hardening relies on changes in solid solubility with temperature to produce fine particles of an impurity phase, (known as second phase particles or precipitates) which impede the movement of dislocations.

From the above results it seems that, During Precipitation hardening hardness was increased gradually up to H1025 condition. From H1075 to H1150 condition hardness is decreased.

The precipitates formed during H1100 and H1150 conditions are having semi- coherency or incoherent with matrix and coarser in size. Due to the incoherency of precipitate with matrix or crystal's lattice, a moving dislocation is then able to bypass the obstacles, by moving in the clean pieces of crystal between the precipitates particles. Thus the hardness was dropped.

The micro structures observed in annealed and precipitation hardened condition is Martensite. Martensite is a supersaturated solid solution of carbon trapped in Body-Centered Tetragonal structure. Martensite is formed due to the faster cooling from above the recrystallization temperature. The transformation is diffusion less. The $M_s$ Temperature cannot be changed by changing the cooling rate. The temperature range of the formation of martensite is characteristic of a given alloy and cannot be lowered by increasing the cooling rate. The $M_s$ Temperature seems to be a function of chemical composition only.

The precipitates formed during precipitation hardening are submicroscopic particles, nanometers (nm) in size. They are invisible under optical microscope. These precipitates are visible under SEM (scanning electron microscope).

## V. CONCLUSION

During solution annealing all the impurities gets dissolved in the solution. Hardening trapped impurities gets precipitated in the form of second phase particles. Due to the impeded motion of dislocations by strong obstacles (precipitates), the strength and hardness are increased. H1100 and H1150 conditions can be noted as over aging conditions, because, the precipitates formed during these conditions are incoherent with matrix and coarser in size. Thus the hardness and strength are decreased. These precipitates are submicroscopic particles, nanometers (nm) in size, Invisible under optical microscope. These precipitates are visible under SEM (Scanning electron microscope).

## REFERENCES

[1]. Military HandBook, MIL-HDBK-6875 1993 *Military specification, heat treatment of steel raw materials*

[2]. Military Standard, MIL-STD-163 1988 *Military standard steel mill products, prepared for shipment and storage*

[3]. Product Data Sheet of 15–5 PH 2007 *Stainless Steel*, West Chester, Ohio: AK Steel Corporation

[4]. Type Approval Document for 15–5 2004 *Precipitation hardening stainless steel*, Mishra Dhatu Nigam Limited

[5]. Type Test Schedule for 15–5 PH 2001 *For forged and hot rolled bars*, RCMA (M)/76

[6]. ASTM E 1019 2003 *Standard test methods for determination of carbon, sulphur, nitrogen and oxygen in steel and in iron, nickel and cobalt alloys*, West Conshonhocken, PA: American Society for Testing and Materials

[7]. ASTM E 112 1996 *Standard test methods for determining average grain size of metals*, West Conshonhocken, PA: American Society for Testing and Materials

[8]. ASTM E 45 1997 *Standard test methods for determining the inclusion content of steel*, West Conshonhocken, PA: American Society for Testing and Materials

[9]. ASTM E 466 2007 *Standard practice for conducting force controlled constant amplitude axial fatigue tests of metallic materials*, West Conshonhocken, PA: American Society for Testing and Materials.