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EFFECT OF DOUBLE AUSTENITIZATION TREATMENTS ON THE MICROSTRUCTURE AND HARDNESS OF 11.7% CHROMIUM AND 1.4% CARBON STEEL (FMU–11)

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Abstract: In this study, the effect of double austenitization (DA) and single austenitization (SA) on the microstructure and hardness of FMU–11 steel, which is used to make the cement mill, was investigated. To obtained maximum hardness; FMU–11 steels are used in a quenched and tempered condition. This involves heating the material to the austenitizing temperature (850–1100ºC), quenching at a suitable rate to form martensite, and tempering to increase the toughness and reduce the retained austenite content. In this investigation, four samples as SA and four other samples as DA was heat–treated at 900, 950, 1000, and 1050 ºC, respectively. The microstructure of the samples was studied using an optical microscope and then scanning electron microscopy (SEM) was applied for higher magnification studies. The hardness measuring for the samples heat–treated in different cycles. The results showed that the DA compared to SA heat treatment will reduce the size of the carbides but will increase retained austenite. However, the hardness decreases as the quenching temperature is increased from 900 to 1050 ºC
Keywords: double austenitization, heat treatment, cement mill, microstructure, hardness

INTRODUCTION

FMU steels are high carbon– high chromium steels that are widely used in cement mills. These steels are air–hardening types with maximum dimensional stability during heat treatment and give high hardness and wear resistance. The heat treatment methods recommended for high chromium martensitic steels continue:

- Austenitization between 950 and 1100ºC followed by quenching in the air;
- Tempering between 200 and 300ºC for high strength, medium–toughness, and resistance also between 600 and 700 ºC for medium–strength, high toughness [1, 2].

An optimum combination of high strength and high toughness in the steel can be achieved only under carefully controlled heat treatment conditions. Earlier studies revealed that austenitizing at a lower temperature of 950ºC did not allow a large number of alloy carbides to go into the solution, leading to the achievement of lower strength and toughness. Moreover, a high austenitizing temperature of 1100ºC or higher, notwithstanding helping in a dissolution of alloy carbides, resulted in an increase in prior austenite grain size besides increasing the δ–ferrite, as well as retained austenite content. Hence, double austenitization (DA) can be applied to obtain the benefits of both treatments. The advantages of DA treatment were reported on other steels [1–9]. The present study has therefore been taken up to evaluate the effects of single austenitization (SA) and Double austenitization (DA) treatments on the microstructure and hardness of FMU–11 steel. The objective of the present study was to explain the mechanisms concerning how the size of the carbides is affected by microstructure through mainly DA treatment for FMU–11 steel.

EXPERIMENTAL PROCEDURE

— Material – specimen preparation

Test samples used in the current study, sectioned with the dimensions of 25 mm × 25 mm ×150 mm, were prepared from a high carbon–high chromium steel (FMU–11) with the chemical composition determined by the Hilger spectrometer, as shown in Table 1. The steel was melted using an induction furnace and then poured into a silica sand mold.

Table 1. Steel Composition.

Element	Composition (%)
C	1.4
Si	0.38
Mn	0.69
Cr	11.71
Mo	0.29
V	0.039

— Heat Treatment

Before the heat treatment, the surface of the samples was coated with cupric sulfate ($CuSO_4$) to prevent oxidation and de–carburization. Due to the low heat transfer coefficient of this steel [10], the samples were heated to 650°C, with a heating rate of 70°C/h and after keeping them there for 30 minutes, they were isothermally treated at different temperatures and times as given in Table 2 and 3. At these temperatures, the samples were kept for 0.5 minutes per mm of thickness, followed by a direct quenching process in the compressed air environment and then tempered in 250°C for 1 h.

— Hardness Testing

The hardness of all the samples measured at 10 Kg minor load and 150 Kg major load using a Vickers hardness tester according to ASTM standard E384 [11].

Table 2. Single Austenitisation (SA)

Sample	Austenitisation	Tempering
T1	900°C	250°C
T2	950°C	250°C
T3	1000°C	250°C
T4	1050°C	250°C

Table 3. Double Austenitisation (DA)

Sample	Austenitisation	Tempering
H1	900°C	250°C
H2	950°C	250°C
H3	1000°C	250°C
H4	1050°C	250°C

— Metallographic Techniques

The light microscopy specimens were prepared based on the ASTM standard E3 [12]. The chemical etchant used to reveal matrix and carbides in hardened steels was 4% Picral. The time of etching varied following different heat treatments. The SEM samples were prepared by mounting in a conductive polymer Polyfast, to minimize the effect of charging.

— Microstructure Imaging

Nikon's high–resolution MA200 Microscope with the camera was used for the examination of the microstructure of specimens. The Tescan Vega–3 LMU a high–resolution scanning electron microscope (SEM) that uses a field–emission electron source was used for imaging.

— Volume Fraction of phases Measurement

The volume fraction of retained austenite and carbides (primary and secondary carbides) in the steel matrix was calculated by using CLEMEX image analyzer software.

RESULTS AND DISCUSSION

— Optical and SEM Microscopy

The optical micrographs of the single heat–treated samples are shown in Figure 1 – T(1–4), and those of double heat–treated shown in Figure 2 – H (1–4). The volume fraction of carbides and retained austenite shown in table 4.

Table 4. Carbide & Retained Austenite Volume Fraction with 2% STD dev

Sample	Carbide Volume Fraction (%)	Retained Austenite (%)
T1	11	4.7
T2	7.9	6.3
T3	5.4	9.5
T4	5.7	11
H1	5.5	9.9
H2	4.7	12.9
H3	4.6	17.1
H4	4.1	19.8

The number of carbides decreased with increasing austenitization temperature from 900 to 1050°C. It was also observed that the DA treatment helped in taking most of the carbides into the solution. The size and number of undissolved carbides were lower in the double treated steel sample compared to the single treated ones. The packet size of martensite laths was also observed to have increased marginally on raising the austenitizing temperature. The retained austenite content increased from 4.7% when austenitized at 900°C to 11% at 1050°C. On DA treatment the retained austenite content of the steel further increased to 19.8%. No significant change in the volume fraction of retained austenite was noticed on tempering the samples at 250°C as compared to the as–quenched condition.

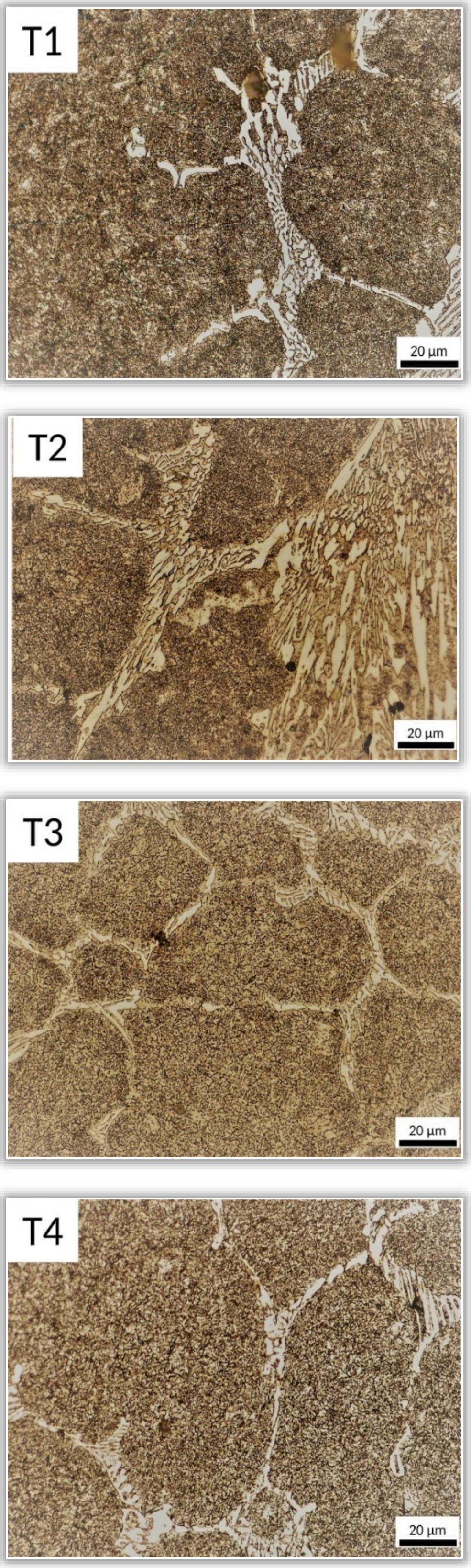

Figure 1. Optical Micrographs of Single Austenitisation T1–T4

The double austenitization treatment helps carbide dissolution. Careful selection of austenitization temperature (single or double) helps maintain a constant volume fraction of retained austenite. The volume fraction of retained austenite increases with an increase in single austenitization temperature and also further increases with the DA treatment. The retained austenite remains stable on tempering the steel at 250°C. Such carbides are decreased after austenitization at 1050°C owing to the higher solubility of carbon in austenite at this temperature. These undissolved carbides provide abundant nucleation sites for austenite nucleation during the second austenitization treatment, resulting in finer carbides.

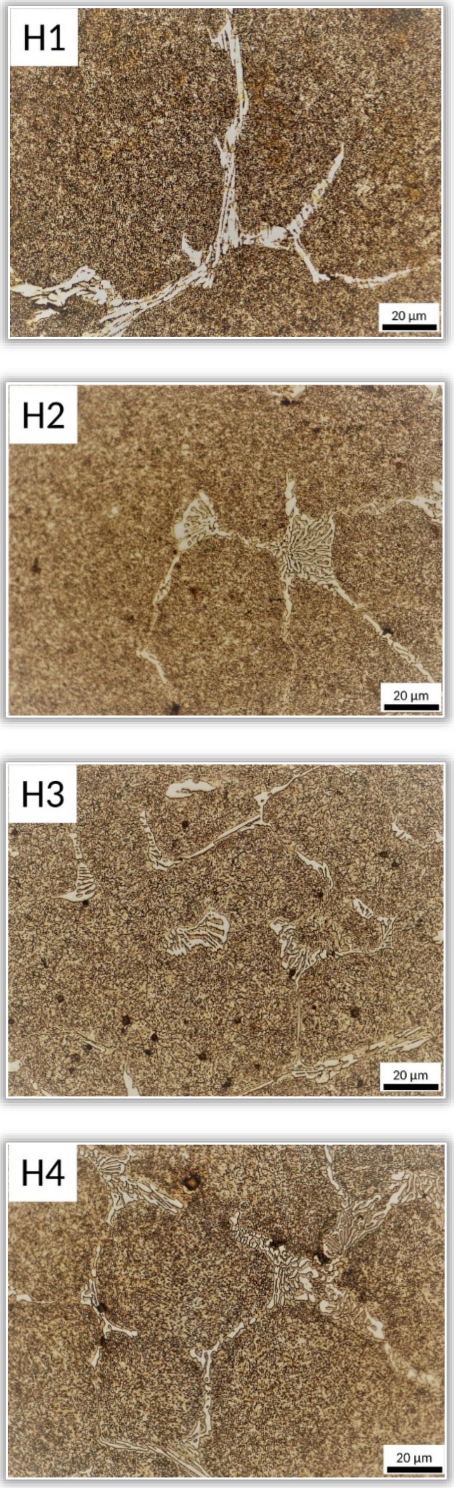

Figure 2. Optical Micrographs of Double Austenitization H1–H3

The SEM images in Figure 3 show that the only difference between the two samples, T3 and H3, was the fine–grained carbides. During the double austenitization at 1000°C, the primary carbides were decomposed and during the cooling and tempering, they were converted into fine–dispersed carbides in the structure. Specimens in both the conditions exhibited typically lath martensite and interlath contiguous films of retained austenite.

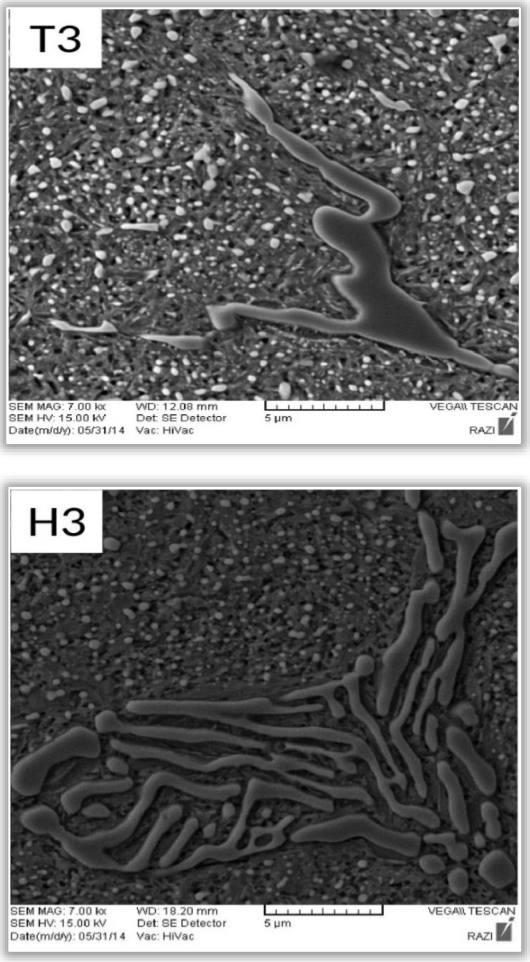

Figure 3. SEM Images of Single Austenitization T3, Double Austenitization H3

— Hardness

On increasing the first austenitization temperature from 900 to 1050°C, the hardness increased from 50 to 58 HRC while in the case of double austenitization the hardness decreased from 58 to 48 HRC. Differences in hardness on tempered samples quenched from various austenitization treatments are given in Table 5. The increase in the hardness with increasing austenitization temperature is because of the rise in the carbon content of the austenite transforms to martensite on quenching. The limited decrease in the H treated samples (compared to that of SA) could be attributed to the slightly higher retained austenite content of the DA treated samples and the decrease in carbon content due to carbide precipitation during second–stage austenitization as was assumed before [1]. Thus the second austenitization in DA temperature should be below the first austenitization (much below solubility of carbides) else it will have a negative effect on hardness.

Table 5. Hardness Values with ±1 STD dev

Sample	Hardness (HRC)
T1	50
T2	56
T3	57
T4	58
H1	58
H2	55
H3	53
H4	48

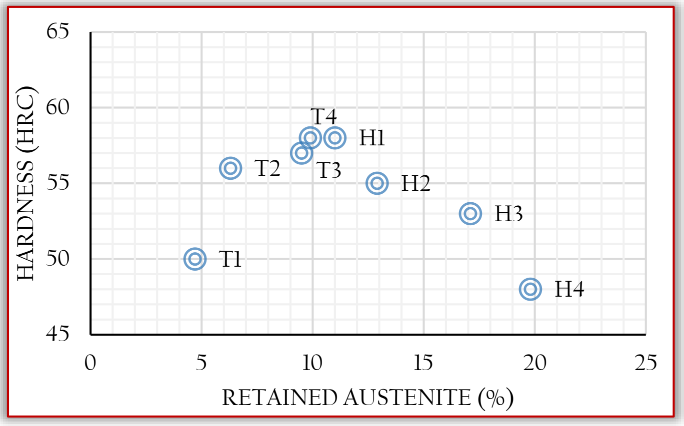

Figure 4. Graph depicting comparison of single and double heat treatments.

CONCLUSIONS

The following conclusions can be drawn based on the present study:

≡ As seen from the graph (Figure 4) of hardness and retained austenite, DA treatment offers relatively better properties as compares to SA treatment.

≡ Undissolved carbides decrease following high temperature (1050°C) single austenitization as compared to low temperature (900°C) single austenitization treatment.

≡ Few undissolved carbides remained in the steel after DA treatment.

≡ The retained austenite increases with an increase in single austenitization temperature from 900 to 1050°C.

≡ Carbide dissolution has a significant effect on grain refinement and hardness. Carbide dissolution in the first austenitization enhanced hardness. An increase in the re–austenitization temperature hurts dissolution of carbides has resulted in a decrease in hardness.

≡ Retained austenite content increases following single austenitization at 1050°C as compared to 900°C, while DA treatment further increases the retained austenite content.

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