



The effect of austenitizing temperature on thenormalising processing behaviour of 34CrNiMo6 steel

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ABSTRACT

The aim of this study is to carry out research about the effect of austenitizing temperature of 34CrNiMo6 low alloy steel on the normalising heat treatment process. Moreover, a comparison of the austenite grain size that formed after the deformation at two austenitizing temperatures of 1100°C and 1200°C with that one resulted after the normalising process at temperature of 860°C. Moreover, to study the effects of increases of holding times of 6, 30 and 60 minutes at normalising process with both austenitizing on the grain size variation, and the consistency of microstructure, which in turn influences on the resulted mechanical properties. It was observed that deformation the specimens at austenitizing temperature of 1100 °C prior normalising process; gives a smaller grain sizes than those when using austenitizing temperature of 1200 °C. Furthermore, Increase in the normalising soaking times does have no influence the increasing of the grain size of austenite. However, it has an effect on The arrangement and shape of grains by making the grains equiaxed and creates a uniform structure. Furthermore, oxides scale thickness was observed at

austenitizing temperature of 1200 °C, and particularly when soaking time at that temperature increased.

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1. Introduction

The 34CrNiMo6 is a low alloy steel, known as a quenched and tempered steel with a characteristic of high hardenability, contains Ni, Cr and Mo. This type of steel is appropriate for thick and large forgings with properties of high tensile strength, as well as impact toughness, and for mechanical parts that usually exposed to high stresses[1]. The production of steel with good homogeneity properties, ductility improvement, and residual stresses reduction can be reach with modifying a series of parameters during deformation process and heat treatment. With increasing the austenitization temperature and soaking time of the prior austenite, grains growth will increase, but with the austenitization temperature the grain growth become more intense[2]. The resulted steel microstructure, which is formed after the deformation process, as well as that formed after heat treatment process, is considerably affected by the austenitizing temperature, where the lower austenitizing temperatures and longer soaking times for normalising give advantages taking into account the austenite grain growth[3]. The objectives of normalising process are refining the grain size, uniformity distributed in the austenitic microstructure[4]. Moreover, S Huang et al[5] concluded that, by extending the holding time of tempering, the martensite will be more recovered and the ferrite resulting from the transformation will be massive. Furthermore, by extend the holding time of tempering; they determined that, the strength of the examined samples started gradually to decrease. According to some researches about the effect of normalising heat treatment process on the both of mechanical properties and final microstructure of the steel[6]. The strength, as well as impact toughness of the 17CrNiMo6 steel can be improved with the refinement in the prior austenite grain size and the packet size [7]. Also E. Boyle [7] mention that the normalising process was effective in refining and homogenizing the grain structure for both steels of 8620 and PS-18 that leads to increase in hardenability. The most important conditions that effect on the oxide scales deforms through the hot rolling process are; thickness of the oxide scale, the temperature of the steel and deformation properties. Matsumoto,

et al.[8]have study the relationship between hot forging and the effect of reduction in friction in steel coated with surface oxide scales. Through the study, they arrived at the conclusion that, existence of oxide scales during the elevated temperature of hot forging process will caused to maintaining the temperature high in steel as result of low thermal conductivity of the surface scales. In another study Okada[9] been investigated the effect of oxide scales on crack initiation in hot rolling deformation of steel. Moreover, the thick oxide scales has tendency to broken and become powder, which in turn will lead to oxide scale defects within the steel products. Furthermore, these defects will initiated the cracks, and these cracks being easier, when oxide scales become thick and rolling temperature increasing.

2. Experimental procedure

The low alloy steel of 34CrNiMo6 is used in this experimental [10]. After the samples were cut and prepared to required shape, the tests were carried out using the servo-hydraulic thermomechanical compression (TMC) machine, which is controlled by a computer system and specifically designed for these tests. The normalising process commenced by heating the specimens up to the selected deformation temperatures of either 1100 °C or 1200 °C, hold it for 10 minutes period of time, deformed using strain of 0.8 and strain rate of 0.5s^{-1} [11], and then being cooled to temperature of 650°C with cooling rate of 5c/sec. Where then the experimental was divided to a two stages. In the first stage, the tested specimens were soaked for a duration of 5 minutes at temperature of 650°C, where then heated up to the normalising temperature of 860°C and soaked for 6 minutes. While, in the second stage the specimens under examination were subjected to a soaking period of 4 hours at temperature of 650°C, as well as soaking durations of 6, 30 and 60 minutes at the normalising temperature of 860°C, consequently. Finally, all the specimens were quenched in tap water to the room temperature as presented in Figure (1). The surface under investigation underwent a series of sequential steps, including grinding, polishing, and etching using a solution of Picric acid to reveal the microstructure. Finally, the resulting surface was ready for metallographic examination by using the optical microscope. Moreover, the MLI (mean linear intercept) method was used to determine the austenite grain sizes.

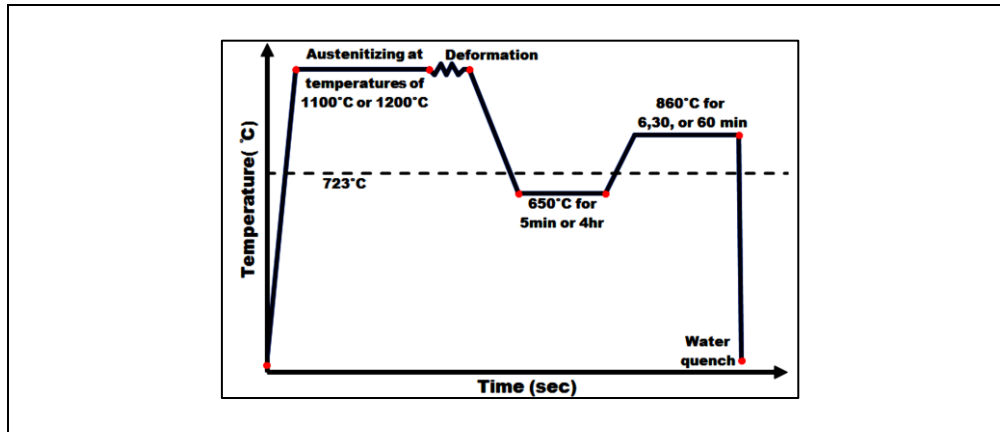


Figure 1 shows the experiment process that consists of austenitizing, deformation, cold down to temperature of 650°C and heated up to 860°C for different soaking times followed by quenching.

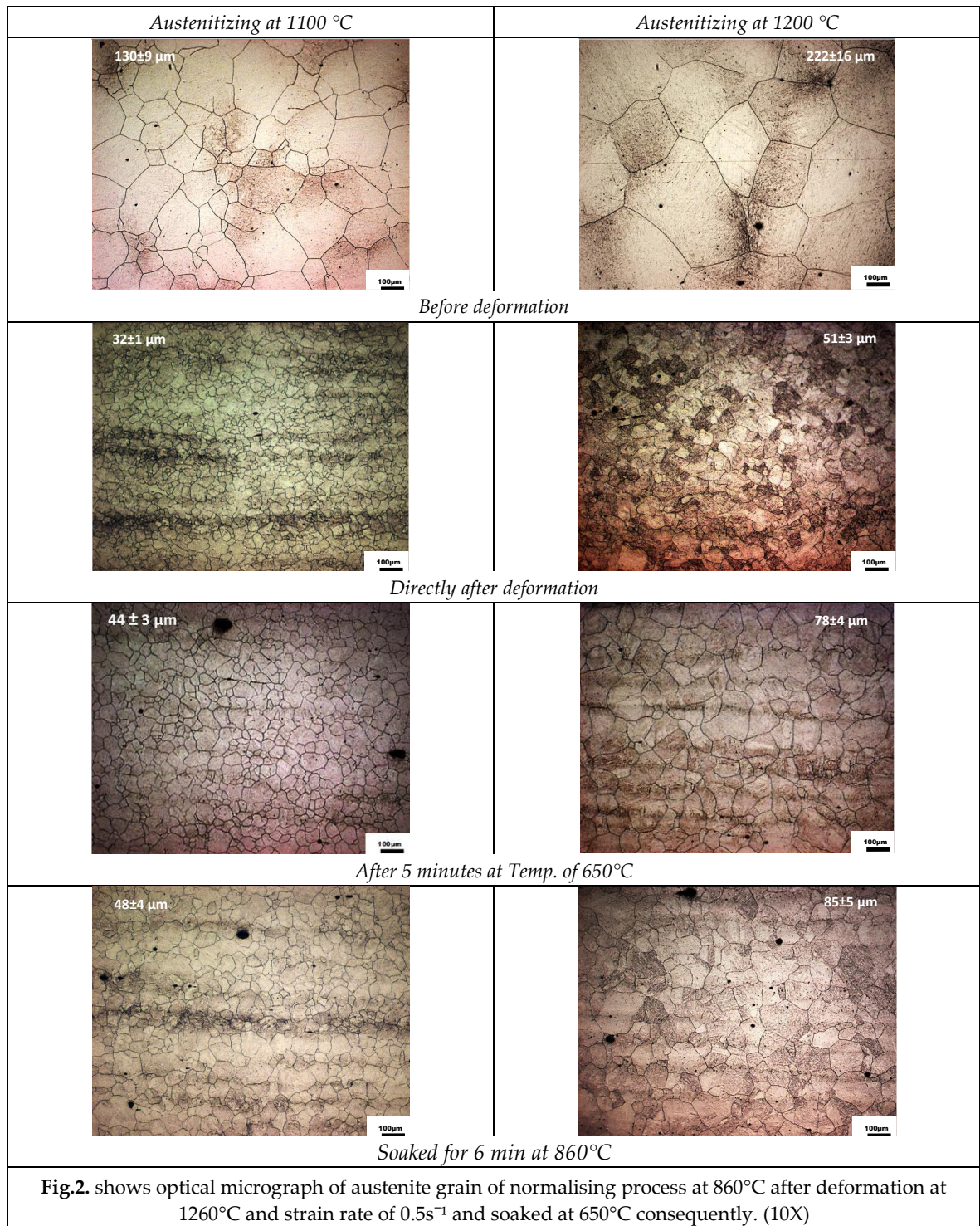
Normally, the oxide scales thickness do not change on the steel surface during the hot forging process at different elevated temperatures and remains the same. However, some oxide scales noticed on the surfaces of some tested samples. Furthermore, to investigate the relation between the thickness of oxide scales, that were observed during doing experiments on the steel surface, and the changing of austenitizing temperature, more test were carried out. Several specimens were positioned in the furnace (muffle furnace) separately to be heated at two different times far apart. Whereas, a times of 1 hour and 24 hours with both of austenitizing temperature 1100 °C to 1200 °C, respectively was used, to examine the effect of these austenitizing temperatures and holding time at that temperatures on the oxide scales.

3. Results and Discussion

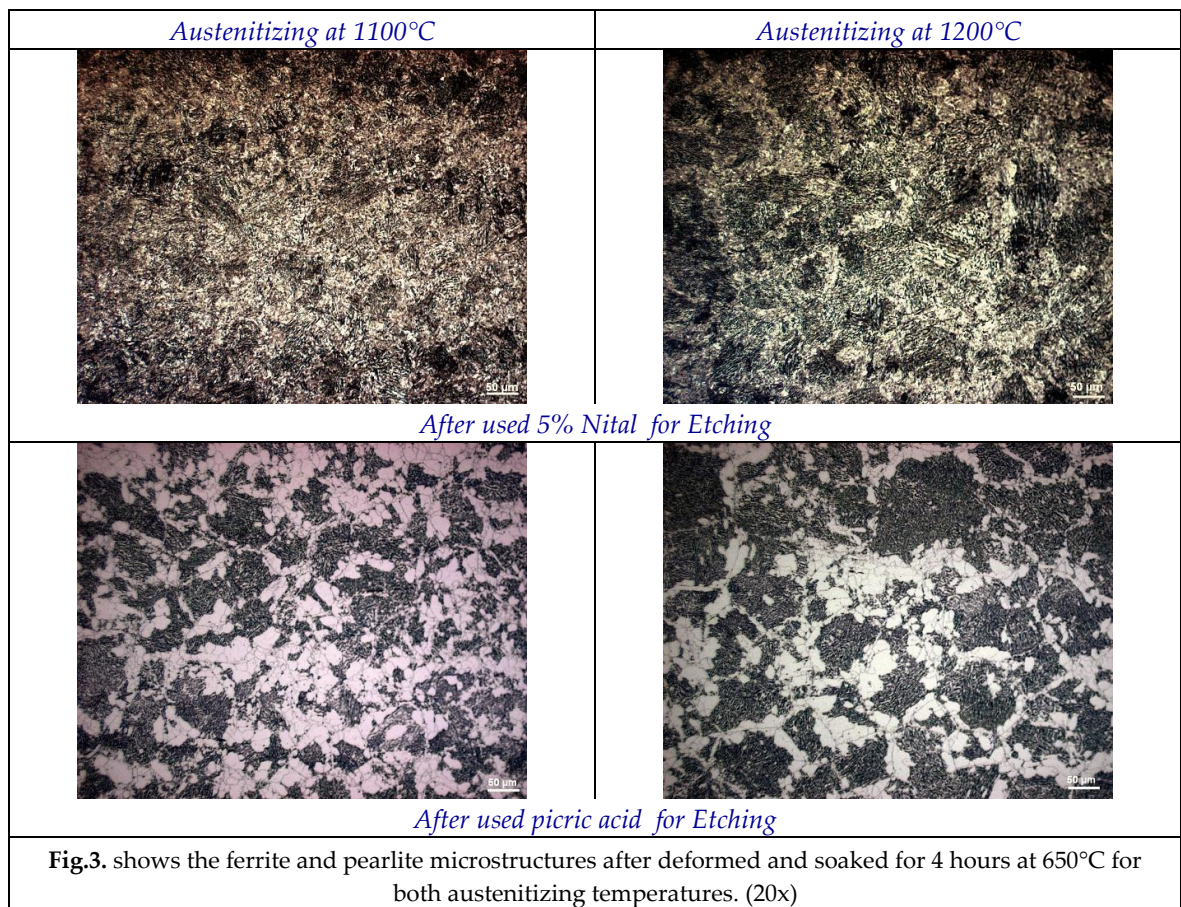
As you can see from the results, it is evident that increasing the temperature leads to the formation of larger grains. The percentage difference in austenite grain size prior to deformation, which was 130 μm and 220 μm and after deformation, which was 32 μm and 51 μm at austenitizing temperatures of 1100 °C, 1200°C respectively, was approximately the same, where it was about 25% and 23%.

At the first stage, after a 5-minute soak at 650°C, there was no observable alteration in the microstructure for both austenitizing temperatures. This is likely because the duration was insufficient to transform the material from its austenitic state to either ferrite or pearlite, and the given the relatively low temperature. Therefore, the average austenite grains size remained nearly consistent both before and after 5 min,

where it was $44 \pm 3 \mu\text{m}$ and $78 \pm 4 \mu\text{m}$ at austenitizing temperatures of 1100°C , 1200°C respectively. While, when the temperature rose to 860°C and soaked for 6-minutes, a minor increase occurred in the size of the austenite grains, where it was $48 \pm 4 \mu\text{m}$ and $85 \pm 5 \mu\text{m}$ at an austenitizing temperatures of 1100°C , 1200°C respectively, as seen in Figure (2).



In the second stage, from the TTT (Time-Temperature-Transformation) diagram for 34CrNiMo6 steel, the structure underwent substantial transformation to consist mostly of ferrite and pearlite following a 4-hour soak at 650 °C. The test specimens underwent etching using a 5% Nital solution, and were also treated with picric acid, as illustrated in Figure (3). As can be seen from the images, the microstructures were clearer when etched with picric acid than Nital. The volume fraction of ferrite and pearlite was approximately commensurate at temperature of 1100°C, while at temperature of 1200°C the grain size of pearlite was very big comparing to the grain size of ferrite, and the volume fraction of pearlite was higher.

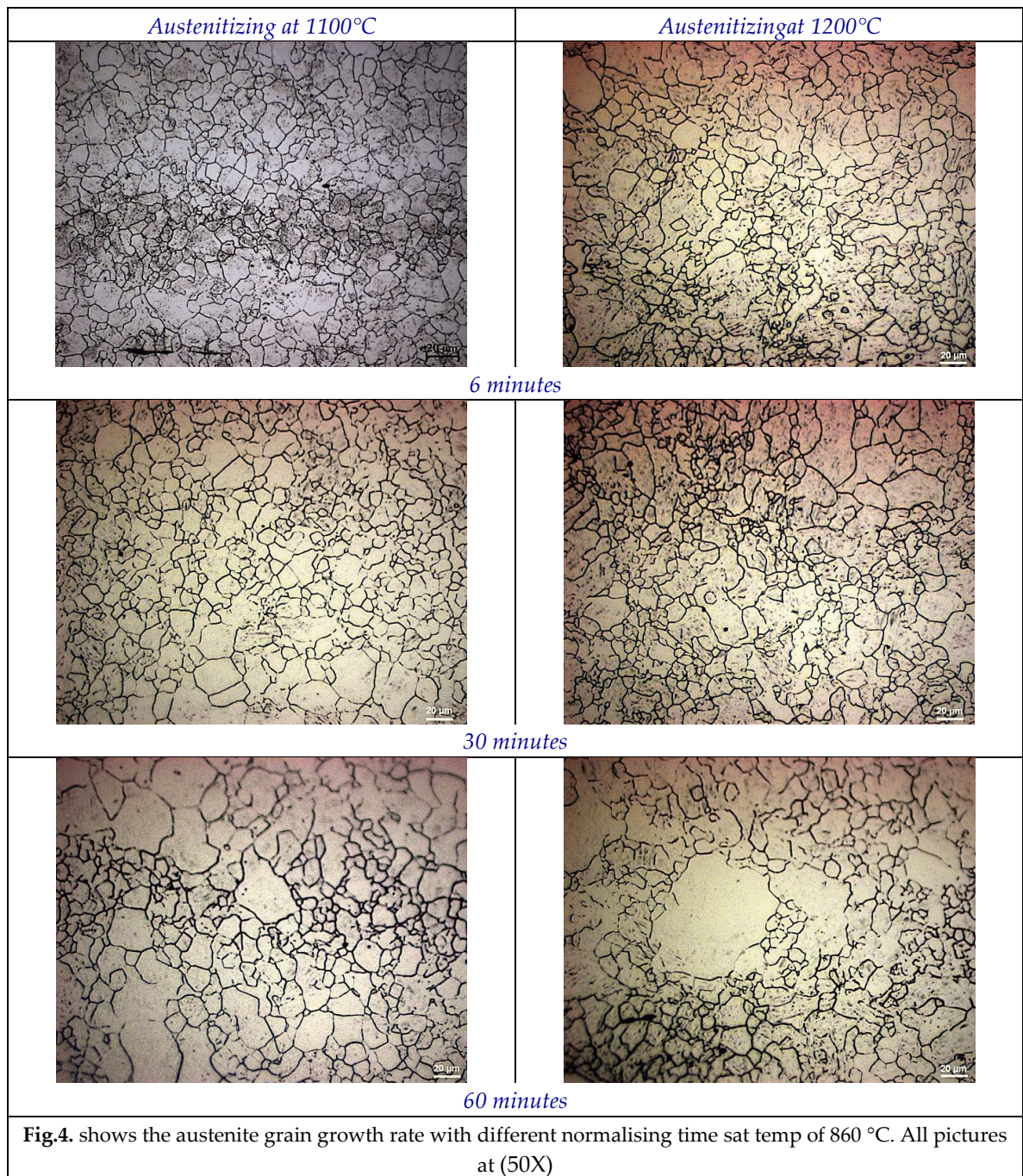


Following that, the temperature was elevated to 860 °C and maintained for a duration of 6-minutes. As a result, new grain nuclei formed, returning the microstructure to austenitic state. As a result, of the 6-minute hold at the normalising temperature of 860 °C, the grain size became refinement, the average austenite grain size became smaller and was reduced to around $9 \pm 1\mu\text{m}$ and $10 \pm 2\mu\text{m}$ at austenitizing temperatures of 1100 °C and 1200 °C, respectively.

As illustrated in Fig. (4), the average grain size was almost remained the same. However, at the austenitizing temperature of 1100 °C, the austenite grains exhibited a more equiaxed morphology with a uniform structure compared to the temperature of 1200 °C, which leads to improving in mechanical properties. After that, when the normalisation time was changed and the soaking time increased to 30-minutes (five times longer), the average size of the prior austenite grains remained relatively unchanged compared to the 6-minute duration, where nearly was $10 \pm 1\mu\text{m}$ and $11 \pm 1\mu\text{m}$. Nevertheless, in terms of microstructure, some slight changes were observed, including some grains exhibiting slight growth, which resulted in reduced uniformity, particularly at the austenitizing temperature of 1200 °C.

Whereas, when extending the duration of the holding time to 60-minutes (ten times longer), there was no significant change on the average of austenite grain size and the grain size was equal for both austenitizing temperatures. There was a minimal increase in the grain size, where it became $12 \pm 2\mu\text{m}$, as seen in Fig. (4), but the substantial change occurred in the grains shape, where two different sizes appeared; small and too big grains (abnormal). An abnormal increase in austenite grain size is noticeable at an austenitizing temperature of 1200 °C; these abnormal grains can potentially lead to a reduction in the material's toughness.

This brings us to that, for both austenitizing temperatures, prolonging the normalisation time did not significantly affect the average size of austenite grains; instead, the size remained relatively unchanged, despite a substantial increase in holding time. This could be attribute to the low normalisation temperature, which may not have been sufficient to promote grain growth, even with longer holding period. In contrast, the optical microstructures of the normalised grains displayed differences in grain shape and distribution, even though the average grain size remained the same.



4. The oxide scale:

Oxidation of steel occurs, when it is exposed to high temperatures in the existence of air. In sometimes, the result of increased oxide layer thickness at elevated temperatures leads to an undesirable oxidation scale. This is due to the potential of the presence or formation of defects like porosity and cracks within the oxide structure.

During the experiments, a presence of oxides scale were revealed these oxides formed on the surface of the test samples when soaked for different times at both

austenitizing temperatures, as seen in Figure (5). These oxide layers became thicker in two cases, when austenitizing temperature increased from 1100°C to 1200°C, and when increasing soaking time at that temperature. Remarkable increase of thickness of oxide scale layer was noted at temperature 1200°C than at 1100°C. Figure (6) Shows SEM images of the oxide scale cross section and EDX, which is used to get for qualitative determination of the elements existing in the selected test sample area.

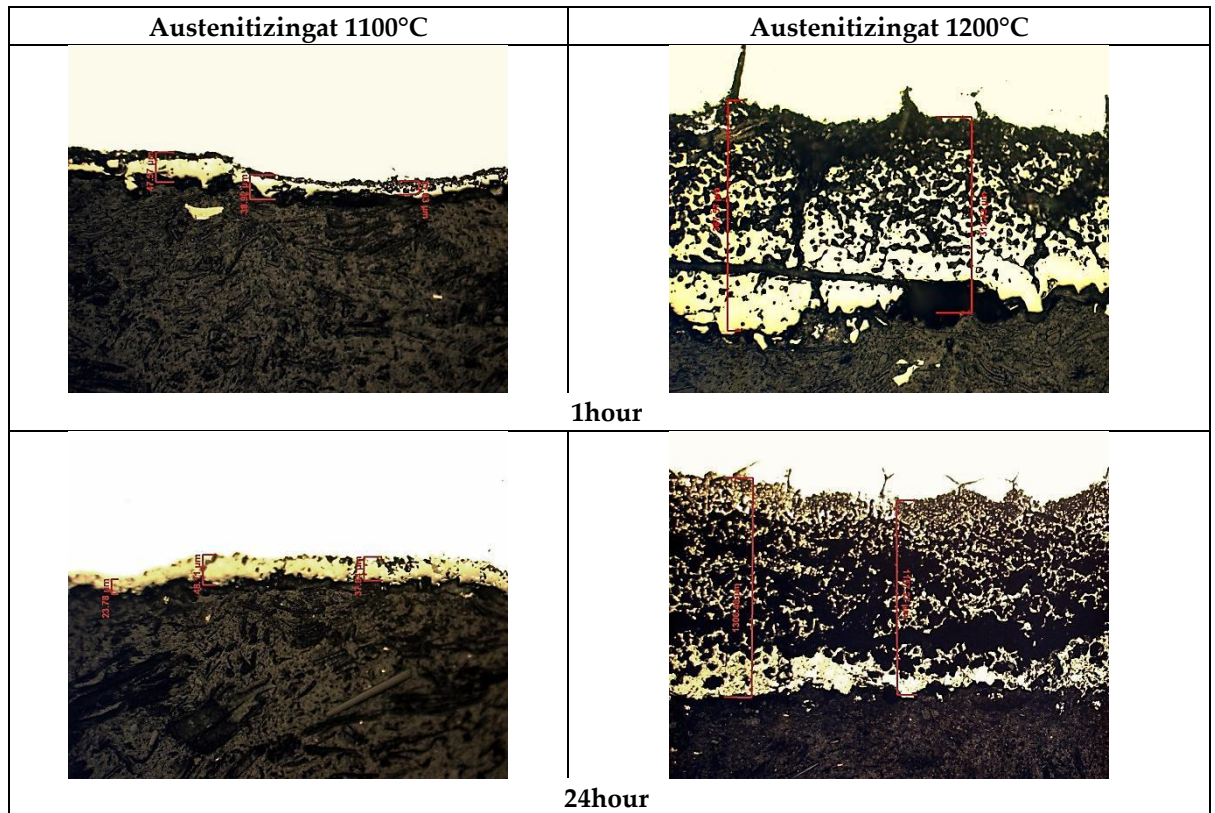


Fig.5. shows the oxide scales that formed on the specimens surfaces at both austenitizing temperatures. All pictures at (20X)

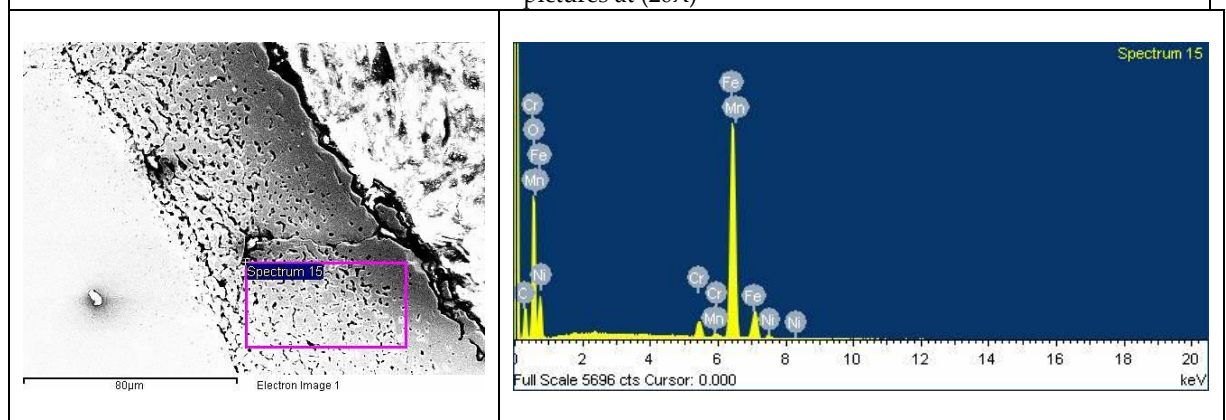


Fig.6. shows SEM image, and EDX microanalysis of the oxide scale for the experiment cross section.

5. Conclusion:

- In the second stage, the prior austenite grain size reduced from 130 μm to 10 μm and from 220 μm to 15 μm after the normalising process for austenitizing temperatures of 1100 $^{\circ}\text{C}$ and 1200 $^{\circ}\text{C}$ respectively. This refinement led to improvements in both the strength and impact toughness of the material.
- The kinetics and rate of grain growth increases at elevated temperature, so using a temperature of 1100 $^{\circ}\text{C}$, gives a finer grains compared to when the temperature is raised to 1200 $^{\circ}\text{C}$.
- According to the TTT diagram for 34CrNiMo6 steel, after being subjected to a holding period of 4 hours at a temperature of 650 $^{\circ}\text{C}$, the structure undergoes almost complete transformation to ferrite and pearlite.
- An Increase of normalising time does not lead to an increase in the average austenite grains size. Despite a notable rise in normalising time from 6 minutes to 60 minutes, the grain size remains roughly unchanged. However, the spent normalising time does affect the grains shape and distribution.
- Increasing the austenitizing holding time to 60 minutes caused in presence of an abnormal austenite grain size particularly at an austenitizing temperature of 1200 $^{\circ}\text{C}$, therefore, to improve the toughness the austenite grain size should be reduced and also eliminate the formation of abnormal grain size.
- Increasing the austenitizing temperature and the duration at that temperature leads to a thicker oxide layer. Consequently, this increase in oxide thickness heightens the likelihood of defects such as porosity and cracks developing within the oxide structure.
- Additional tests recommended carrying out to investigate the reasons for changing of oxide scales thickness on the 34CrNiMo6 steel surface with changing of austenitizing temperatures during the hot forging process.

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