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Evolution of Grain Size in 34CrNiMo6 Steel as a Function of Thermo-mechanical Process Route

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Abstract

The main purpose of the isothermal deformation processes is to quantify the austenite grain size at different deformation conditions and determine the stress strain behaviour of the 34CrNiMo6 low alloy steel. In this study we have simulated the deformation process conditions experienced in the large scale forging of 34CrNiMo6 low alloy steel in order to understand and optimize the thermo-mechanical process parameters that determine microstructure and thus the final mechanical properties. Isothermal hot working tests over the temperature range of 900°C - 1260°C, strain rates of 0.1, 0.5 and 1s^{-1}, and strains of 0.4, 0.6 and 0.8, while a range of microscopy methods will be used for microstructure analysis. The peak stress and peak strain for initiation of dynamic recovery (DRV) and recrystallization (DRX) at different temperatures and strain rates were calculated. The relation between grain size diameter and both of Zener-Hollomon parameter and deformation temperatures with changing of strain rate at both austenitising temperatures of 1100°C and 1260°C were determine. All these data are going to be used through the general constitutive equations to determine the hot working constants.

Keywords: *Grain Size, Environmental, 34CrNiMo6, Thermo-mechanical process, Zener-Hollomon parameter .*

1. Introduction

The worldwide demand for increasing energy efficiency in power generation or for drilling for oil in deeper seas has necessitated the manufacture if bigger sections, [1, 2]. In general metal forming processes, such as forging of steels, attempt to improve a desired microstructure, which leads to improve mechanical properties, at the same time as producing a near-net shape, which for large sections can only be done at elevated temperature [3]. The 34CrNiMo6 is a quenched and tempered low alloy engineering steel with a high hardenability, containing nickel, chromium and molybdenum. It has good toughness properties with CVN (Charpy V-notch) values averaging around 80J. Thus is an ideal material for large forgings where high tensile strength properties and impact toughness are a requirement [4].

The microstructure, which is formed after the deformation or after heat treatment processes, is significantly affected by the austenite grains size, where a small austenite grain size is preferred to improve impact toughness and strength. Many investigators have reported that the prior austenite grain size plays a significant role in controlling of some mechanical properties of steels. For instance, the strength and impact toughness of the 17CrNiMo6 steel can be improved with the refinement in the prior austenite size[1]. Moreover, The refinement of martensitic structure was obtained by reducing the quenching temperature from 1200°C to 900°C, and the refinement of prior austenite grains can prompt the refinement of martensitic packets and blocks, which resulting in enhanced of the yield strength and toughness simultaneously [2, 3].

2. Experimental procedure

The chemical composition of the 34CrNiMo6 low alloy steel used in this work is given in table (1). The specimens were austenitised either at 1260C° or 1100C° for one hour (according to the holding time estimation). After that, the specimens were cooled in 60sec to a particular deformation temperature. The one hit of isothermal deformation process takes place, using a strain of 0.8 with strain rates of 0.1 s^{-1}, 0.5 s^{-1} and 1 s^{-1} at each deformation temperature of 900°C, 1000°C, 1100°C or 1200°C, respectively after austenitised at 1260°C, and deformed at 900°C, 950°C, 1000°C, and 1100°C, respectively after austenitised at 1100°C. Finally, the specimens were water quenched to room temperature as shown in figures (1).

Table 1: Chemical composition (wt %) of 34CrNiMo6 alloy steel

Element	**Acceptable W% range**	**Measured W%**
C	0.33-0.35	0.33
Cr	1.45-1.7	1.52
Ni	1.6-1.7	1.66
Mo	0.2-0.3	0.23
Mn	0.4-0.7	0.51
Pmax	0.015	0.009
Si	0.15-0.35	0.22
Smax	0.01	0.01
Almax	0.01	0.004

V	0.05-0.12	0.07
Cumax	0.3	0.13
Snmax	0.05	0.01

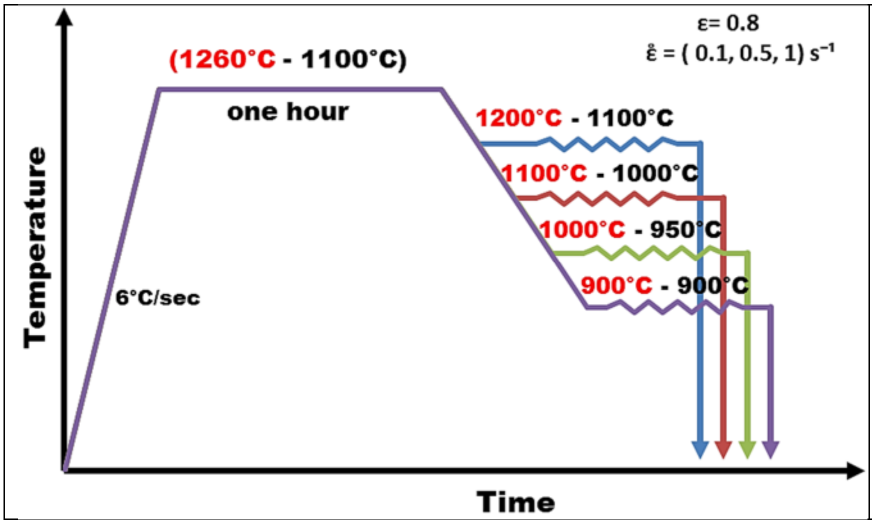

Figure 1. schematic diagram of the isothermal thermo-mechanical processes, using a strain of o.8 with different strain rates (0.1, 0.5 and $1s^{-1}$) at reheating temperatures of (A) 1260°C, at each deformation temperature (900°C, 1000°C, 1100°C and 1200°C), (B) 1100°C, at each deformation temperature (900°C, 950°C, 1000°C, 1050°C and 1100°C).

3. Results and discussion

1.1. Austenitising temperatures and holding time

2. The austenite grain growth is affected by both the austenitising temperature and the holding time, but the effect of austenitising temperature is more strongly than the holding time[4]. The autenitising temperature of 1260°C was used to try to optimize the thermo-mechanical process parameters. To attempt to refinement austenite grain size, the austenitising temperature of 1100°C also was used, to make a comparison to investigate the effect of this change on the austenite grain size. Moreover for all experimental specimens, one hour was the holding time for both reheating temperatures of 1260°C and 1100°C as shown in figures (2, 3). Where the biggest grain size was got with the suitable holding time, while after this time the increase in grain size will be slightly small compared to the bigger increase in heating time. To try to simulate a real large scale forging process for 34CrNiMo6 steel by holding the samples for a long period of time in the austenitising temperature before the deformation process.

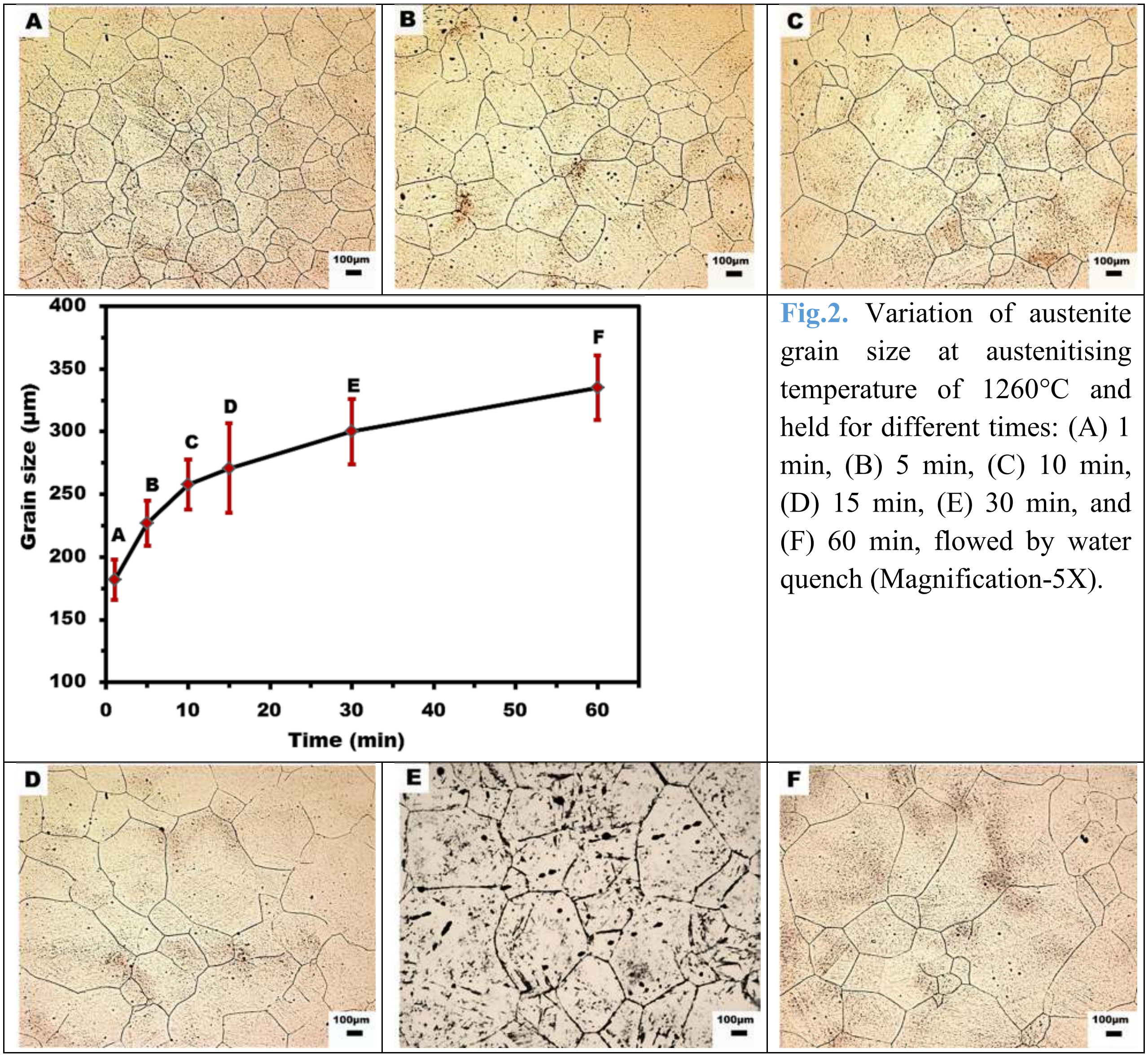

Fig.2. Variation of austenite grain size at austenitising temperature of 1260°C and held for different times: (A) 1 min, (B) 5 min, (C) 10 min, (D) 15 min, (E) 30 min, and (F) 60 min, flowed by water quench (Magnification-5X).

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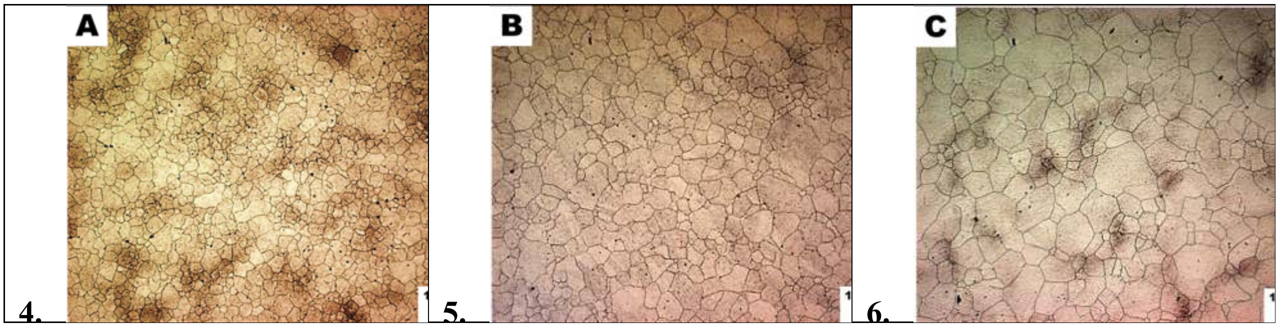

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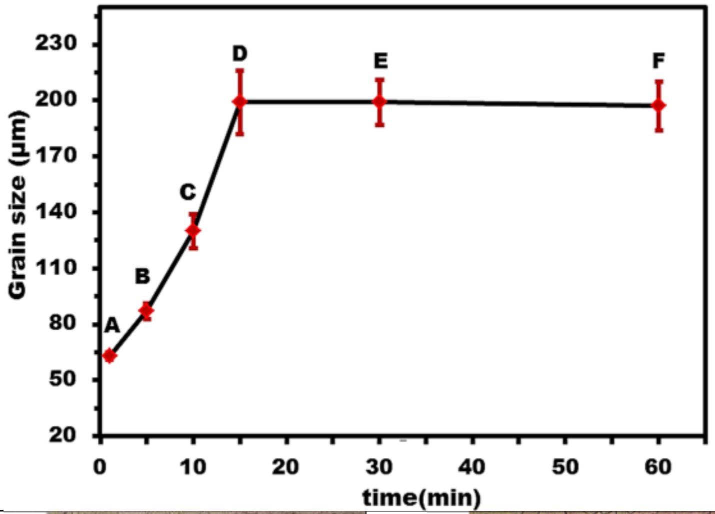

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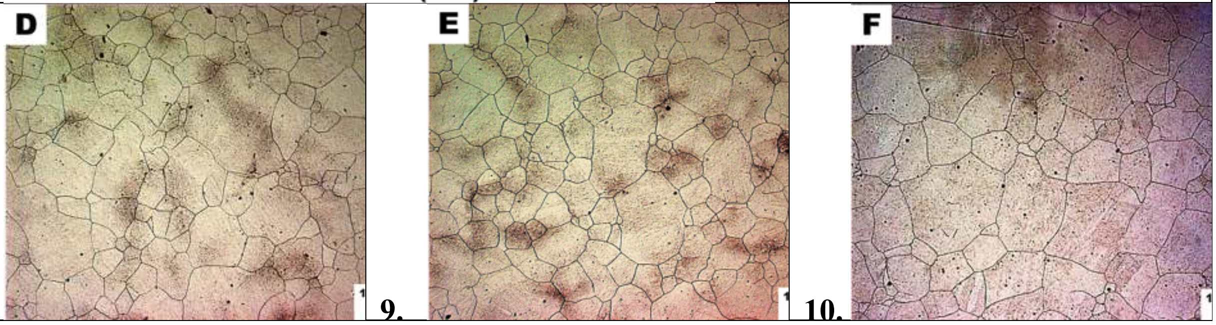
Figure.3. Variation of austenite grain size at austenitising temperature of 1100°C and held for different times: (A) 1 min, (B) 5 min, (C) 10 min, (D) 15 min, (E) 30 min, and (F) 60 min, flowed by water quench (Magnification-5X).



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3.2 . Flow curves and optical microstructure at austenitising temperatures of 1260°C and 1100°C

Figure 4(a)-(f) shows the true stress–strain curves of the 34CrNiMo6 low alloy steel at two austenitising temperatures followed with different deformation temperatures, 0.8 strain, and different strain rates. The effects of deformation temperatures and strain rates can be seen in all curves, where the lower deformation temperature the higher flow stress was obtained, at the same time at the higher strain rate the higher flow stress was achieved. For the reason that, high temperatures and low strain rates gives longer time for energy accumulation and higher ability to move at grain boundaries for the nucleation and growth of dynamically recrystallised grains and dislocation faded. Therefore, flow stress is decreases at low strain rates and high temperatures [4].

A single peak was observed in all the flow stress curves. In the case with austenitising temperature of 1260°C, as can be seen in figure 4(d)-(f), the flow stress increased to the peak and then slightly decreased to a steady state accompanied by dynamic recrystallisation at all deformation temperatures and strain rates. Where in this case, when the stress reach its peak value dynamic recrystallisation softening take place and the peak flow stress slowly decrease, after that hardening and recrystallisation softening balance and the flow stress curve reached to steady state. Excluded from that the deformation temperature of 1000°C with strain rates of 0.5 s^{-1}, and 1 s^{-1} and the deformation temperature of 900°C with all strain rates, where the flow stress increased to a peak and then continuous increasing to reach the steady state in the end. The reason for that may be the return to the resulting massive elongated grains with small percentage of recrystallized grains especially at deformation temperature of 900°C which is approximately the recrystallisation stop temperature. The percentages of recrystallisation at 900°C approximately were approximately 11%, 6% and 5% at strain rates of 0.1, 0.5 and 1S^{-1} respectively.

The peak stress and peak strain with various influencing factors are shown in Table (2). The peak flow stress increases with increase of deformation strain rate, where for example at the deformation temperature of 1000°C the peak stress was 88, 110, and 124 MPa when deformation strain rate was 0.1, 0.5 and 1 S^{-1}, respectively. Also, the peak flow stress increased with decreased deforming temperature, where for example at the strain rate of 0.1 S^{-1} the peak stress was 44, 57, 88 and 145 MPa when deformation temperature was 1200, 1100, 1000 and 900 °C, respectively. In addition, increasing of strain rate and decreasing of deformation temperature will cause delay of the beginning of dynamic recrystallisation. As shown in the table (2) the peak strain ranges from 0.26 to 0.44.

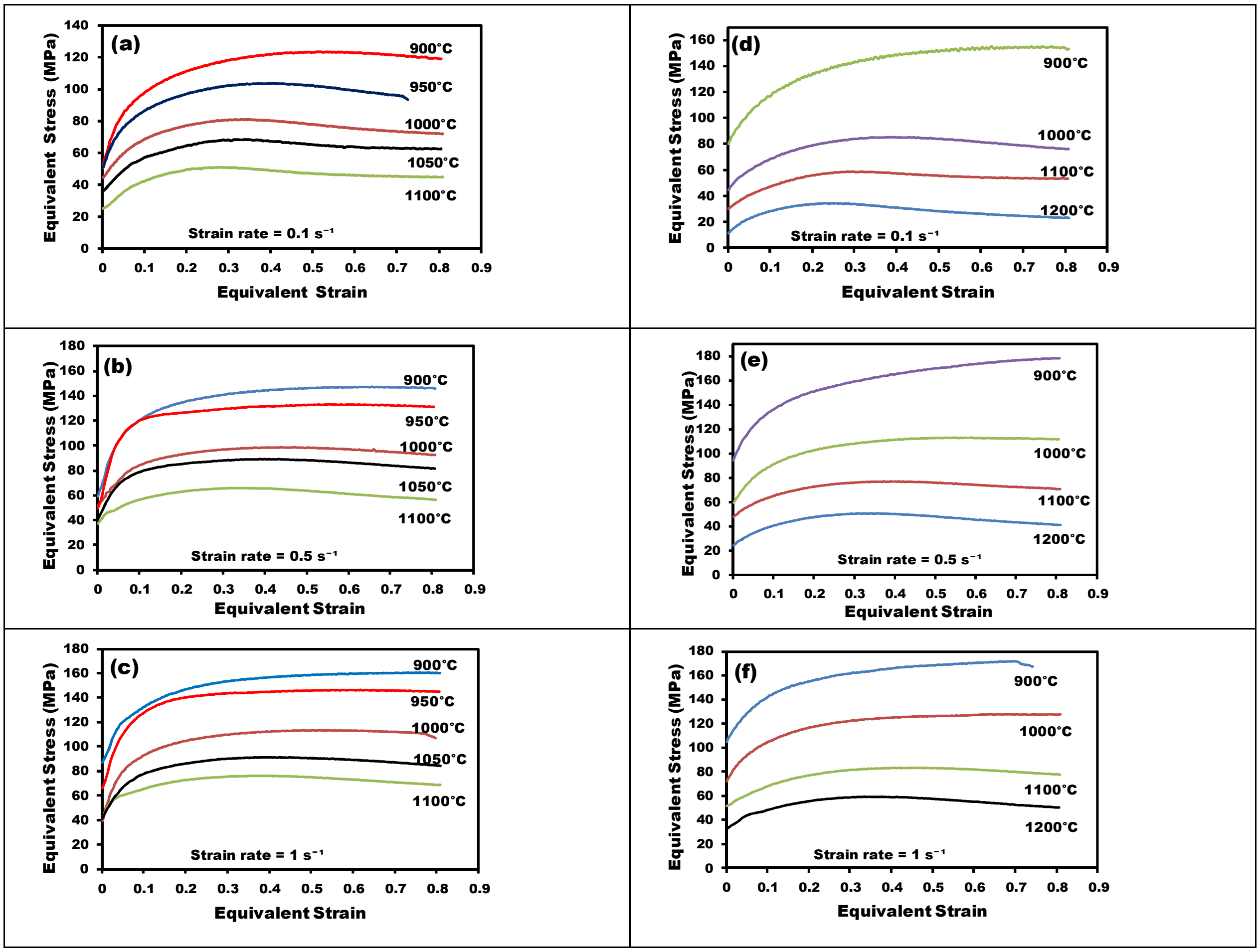


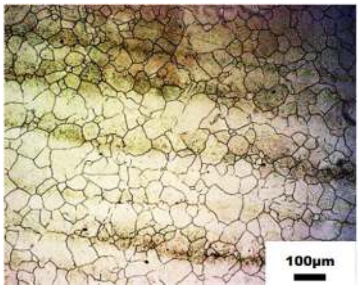
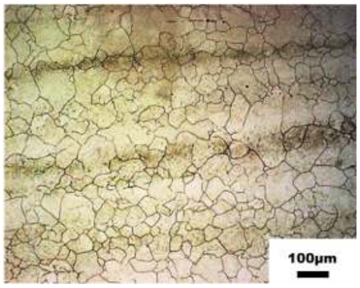
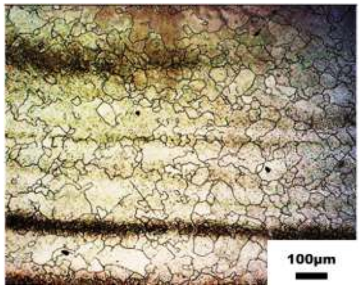
Fig.4. The true stress-true strain curves, after austenising for one hour [(a, b, c) at temperature of 1100°C, and (d, e, f) at temperature of 1260°C] followed by deformed at various temperatures, 0.8 strain and Strain rates of (a, d) 1 S^{-1}, (b, e) 0.5 S^{-1}, and (c, f) 0.1 S^{-1}.

Table 2: Shows the peak stress, and peak strain with different deformation temperatures and strain rates.

Austenitising temperature (C°)	$\mathring{\varepsilon}$ (s^{-1})	Deformation temperature	**εp**	**σp** (MPa)
1260	1	1200	0.34	64
		1100	0.42	84
		1000	0.64	124
		900	0.70	167
	0.5	1200	0.31	56
		1100	0.37	77
		1000	0.59	110
		900	0.8	170
	0.1	1200	0.26	44
		1100	0.32	57
		1000	0.44	88
		900	0.77	145
1100	1	1100	0.39	76
		1050	0.37	91
		1000	0.50	113
		950	0.62	147
		900	0.78	161
	0.5	1100	0.34	66
		1050	0.39	90
		1000	0.44	99
		950	0.56	133
		900	0.66	147
	0.1	1100	0.28	51
		1050	0.34	68

		1000	0.34	81
		950	0.40	104
		900	0.52	124

While in the case with austenitising temperature of 1100°C, as can be seen in figure 4(a)-(c), the true stress-strain curves in general the characteristics of the flow stress curves at temperatures 900°C, 950°C, 1000°C, 1050°C and 1100°C, approximately are similar to the flow stress curves with austenitising temperatures of 1260°C. At strain rate of 0.1 S^{-1} almost in all flow stress curves a single peak was observed, where the flow stress increased to the peak and then slightly decreased to a steady state. Furthermore, at 0.5 and 1 S^{-1} a single peak of flow stress curve was observed, apart from the case of 900°C and 950°C with both strain rates, and 1000°C with just 1 s^{-1} strain rate, where the flow stress increased to a peak and then continuous increasing to reach the steady state in the end. In this case, the dynamic recovery occurred where the work hardening dominant at the beginning of deformation that makes the flow stress increased. Then hardening and softening achieved balance when deformation reached a certain degree, flow stress curve reached steady state. Because of some alloying elements in the chemical composition of experiment steel, the dynamic recrystallisation is retarded. Consequently, the steel has a high recrystallisation temperature. As the deformation temperature is 900°C with the strain rates of 0.1, 0.5 and 1 s^{-1} for both austenitising temperatures as seen in figures (5) and (6), there are some nuclei germinated dynamically on the original grains boundary of deformed grains. The percentages of recrystallisation at deformation temperature of 900°C with austenitising temperature of 1100°C were approximately 24%, 15% and 23% at strain rates of 0.1, 0.5 and $1S^{-1}$, respectively. These percentages are higher comparing with that ones at the same deformation temperature and austenitising temperature of 1260°C.

	1 s^{-1}	**0.5 s^{-1}**	**0.1 s^{-1}**
1200° C			

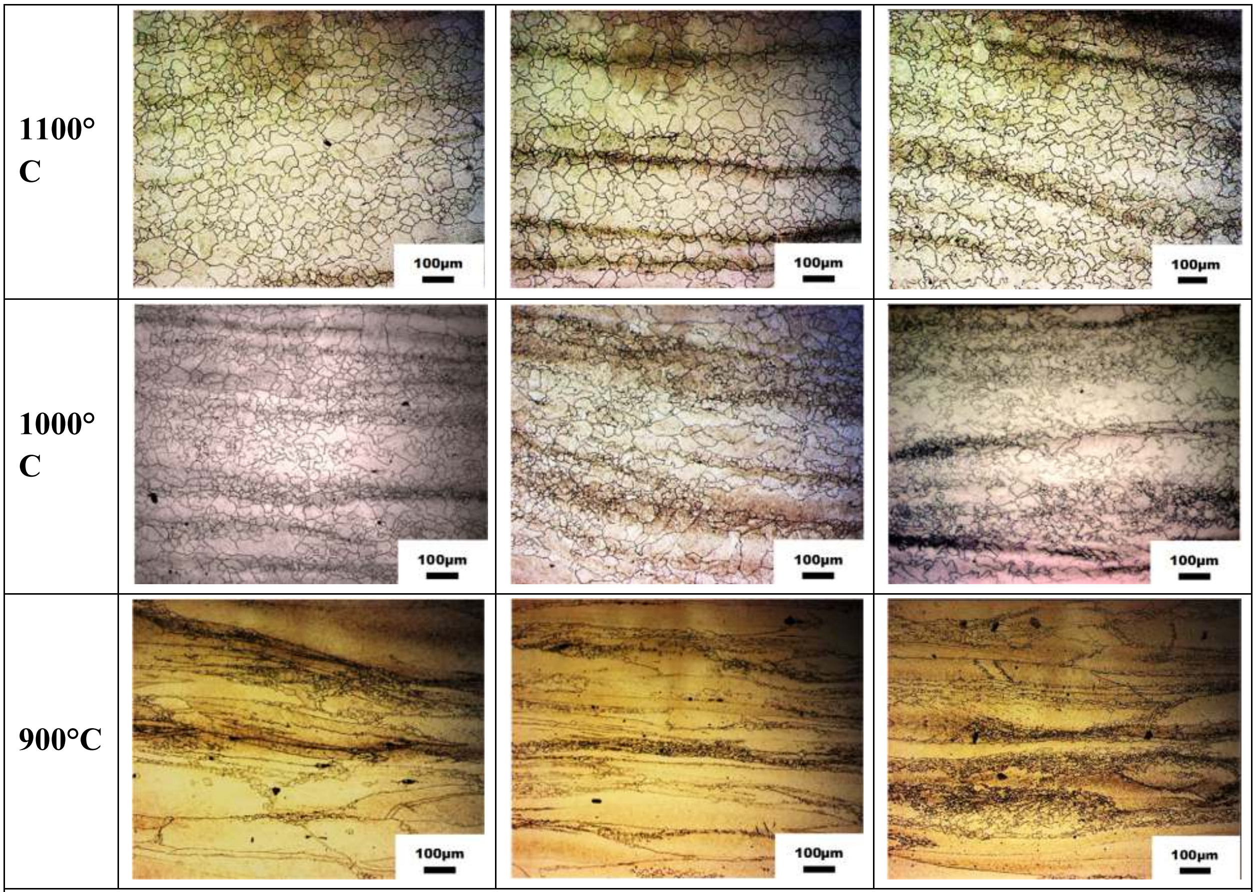


Fig.5. Variation of austenite grain size after austenitising for one hour at temperature of 1260°C and deformed at different temperatures and different strain rates followed by water quench (10X).

3.3 . The relation between grain size diameter and deformation temperatures

The relationship between the grain size and deformation temperatures with changing of strain rate at both austenitising temperature are shown in figure 7 (a, b). As can be seen at reheating temperature of 1100°C, there was a difference in grain size accompanied with change in the strain rate almost at all deformation temperature where the most of the small grain sizes was obtained with strain rate of 0.5 S^{-1}. Whereas, at reheating temperature of 1260°C, the difference in grain size was just at deformation temperature of 1200°C, while at deformation temperatures of 1000°C and 1100°C there was no big difference and the grain size nearly the same.

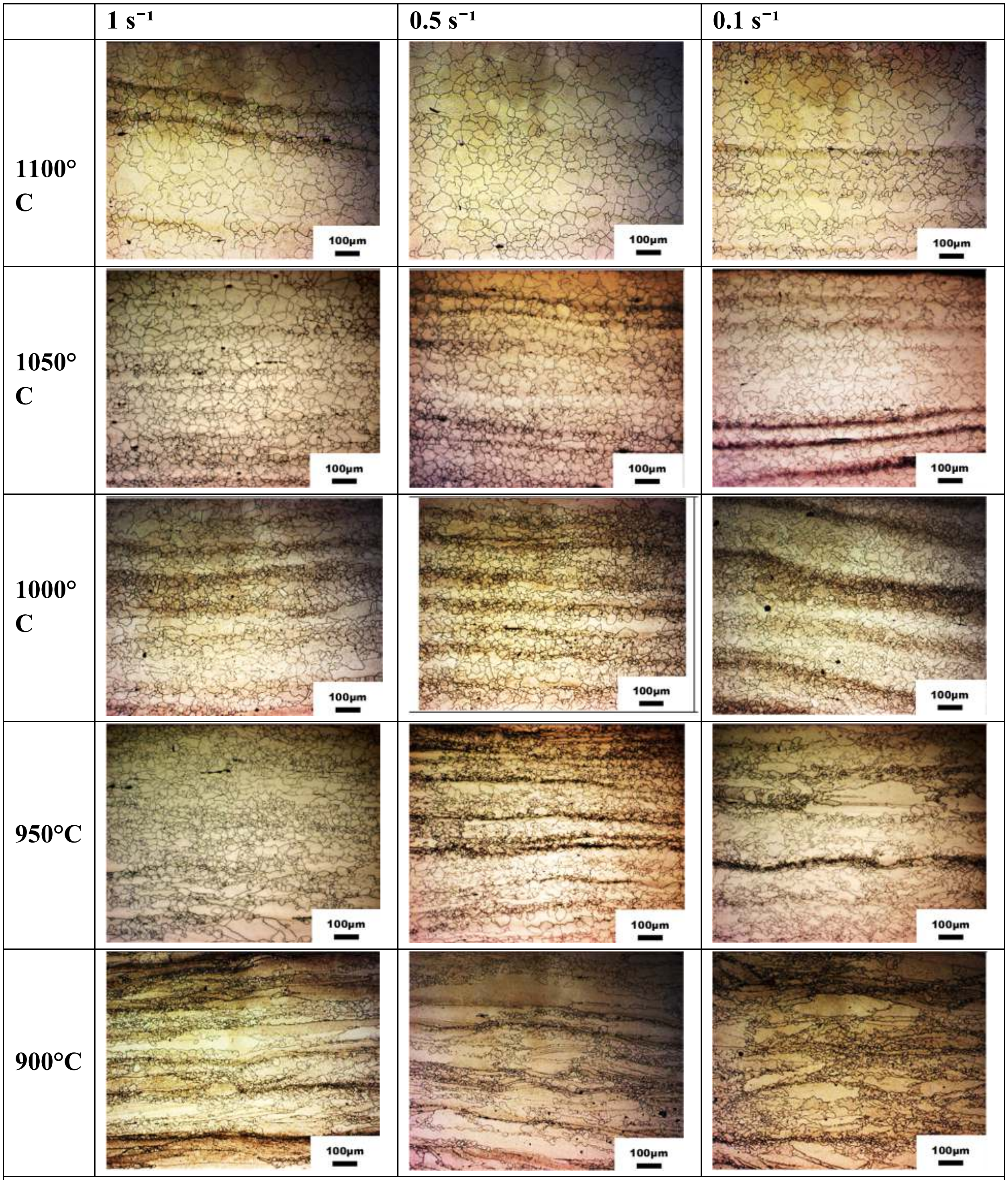


Fig.6. Variation of prior austenite grain size after an austenitising for one hour at temperature of 1100°C and deformed at different temperatures and different strain rates followed by water quench (10X).

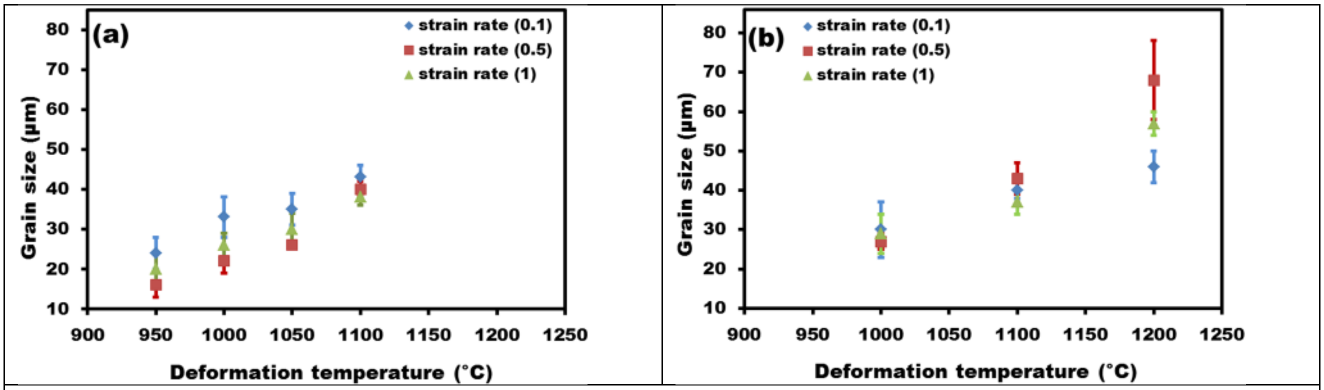


Fig.7. The relation between grain size and deformation temperatures at an austenitising temperature of (a) 1100°C, (b) 1260°C.

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3.4 The relation between grain size diameter and Zener-Hollomon parameter

The main influence factors on Zener-Hollomon parameter (Z) are the strain rate and deformation temperature, but in manufacture process, the equivalent strain rate generally depends on manufacturing equipment. The strain rates of 0.1, 0.5, and 1 S^{-1} were used with all deformation temperatures. From the figure (8), the smaller Z was got with bigger grain size and that's happening with decrease of strain rate and increase of deformation temperature which facilitate the recrystallisation. Inversely, the bigger Z resulted with smaller grain size, and that's happening with increase of strain rate and decrease of deformation temperature, that causes facilitate of the dynamic recovery.

From linear regressions of the results in figure (8), a good relation between grain size and the natural logarithm of Zener-Hollomon parameter was got with reheating temperature of 1100°C, which represented in equation [d=-4.1ln(Z)+173]. Whereas the linear regressions of the results at reheating temperature of 1260°C was lower because of the data slightly scattered at higher temperature of 1200°C, which represented in equation [d=-4.2lnZ+187].

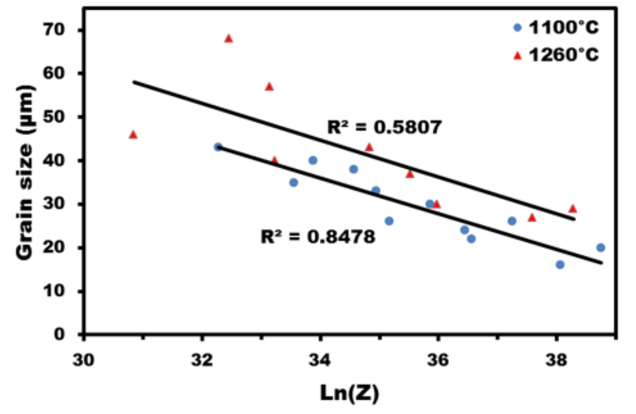


Fig.8. shows the relationship between the grain size diameter and the Ln(Z) in the isothermal processes with one hour reheating temperature of 1100°C, 1260°C and deformed at different temperatures and different strain rates.

3.5 Flow curves and optical microstructure at different strains:

The true stress–strain curves of isothermal processes austenitising for one hour at temperature of 1260°C and cooled down to deformed at temperature of 1000°C with strains of 0.4, 0.6, and 0.8 and strain rates of 0.1, 0.5, and 1 S^{-1} with each strain, as shown in Figures (9). The flow stress curve with strain rate of 0.1 S^{-1} a single peak was observed, where the flow stress increased to the peak and then slightly decreased to a steady state. While at strain rates of 0.5 and 1 s^{-1} the flow stress increased to a peak and then continuous increasing to reach the steady state in the end. The percentages of recrystallisation at strain of 0.6 and strain rates of 0.1, 0.5 and $1s^{-1}$ were approximately 41%, 38% and 37% respectively, as shown in figure 10 (b, e, h). While at strain of 0.4, the recrystallisation occurred with percentages less than that occurred at strain of 0.6, where the percentages at strain rates of 0.1, 0.5 and $1S^{-1}$ were approximately 29%, 26% and 24% respectively, as shown in figure 10(a, d, g).

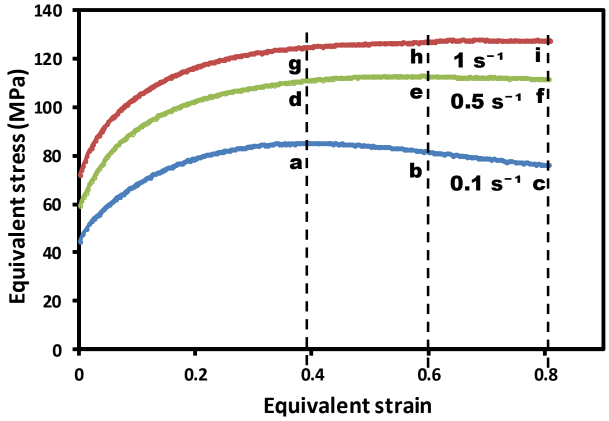
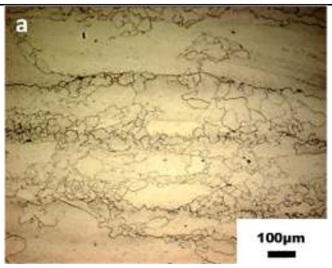
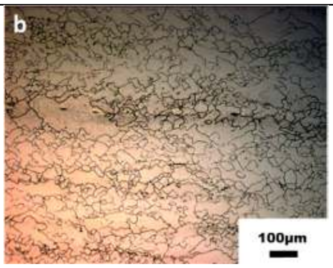
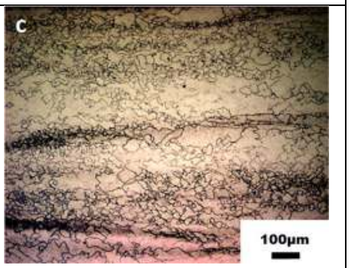
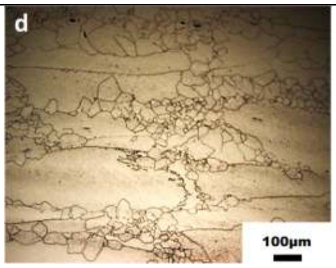
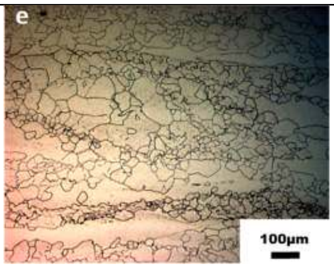
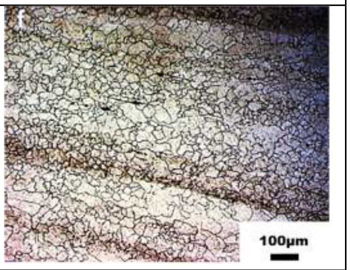


Figure.9. True stress – true strain curves with one hour reheating temperature at 1260°C and deformed temperature of 1000°C and strains of 0.4, 0.6, 0.8 with each strain rates of 0.1, 0.5 and $1s^{-1}$.

	0.4	0.6	0.8
$0.1s^{-1}$			
$0.5s^{-1}$			

1s^{-1}

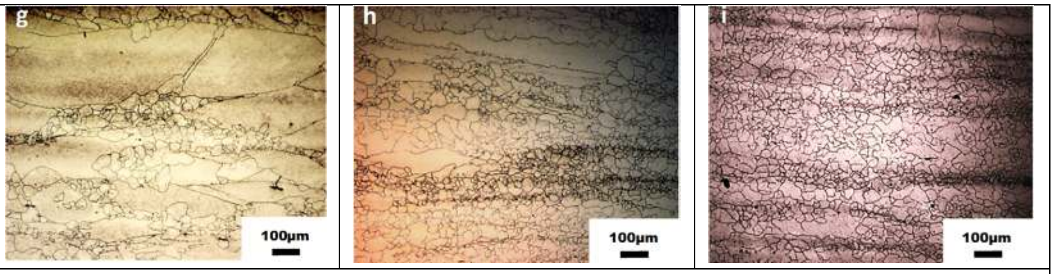


Figure.10. Shows optical micrograph of samples deformed at 1000°C after soaked for one hour at 1260°C, with strain of (a, d, g) 0.4, (b, e, h) 0.6, (c, f, i) 0.8 with strain rates of 1 S^{-1}, 0.5 S^{-1} , and 0.1 S^{-1} (10X).

4. Conclusions

- The fine and equiaxed grains of material lead to improvement in toughness as well as strength. So, at all isothermal processes with two reheating temperatures, the investigation shows that, using an austenitising temperature of 1100°C gives better equiaxed grains and higher recrystallisation percentage than using an austenitising temperature of 1200C°.
    - The smallest grain sizes were observed at an austenitising temperature of 1100°C and strain rate of 0.5 S^{-1} with all consequent deformation temperatures.
- The steel has a high recrystallisation temperature, and 900°C is approximately the recrystallisation stop temperature where some nuclei germinated dynamically on the original grains boundary of deformed grains, thus in order to refine grain size the thermomechanical processing must be performed above this temperature.
- At deformation temperature of 900°C with an austenitising temperature of 1100°C, the percentage of recrystallisation were approximately 24%, 15% and 23% at strain rates of 0.1, 0.5 and 1S^{-1} respectively, which were higher when compared with the same percentage at an austenitising temperature of 1260°C, which reduced to approximately 11%, 6% and 5% at the same strain rates respectively.
- A good relation between grain size and the natural logarithm of Zener-Hollomon parameter was got with reheating temperature of 1100°C, While the linear regressions of the results at reheating temperature of 1260°C was lower because of the data slightly scattered at higher temperature of 1200°C.
- The percentages of recrystallisation at strain of 0.6 and strain rates of 0.1, 0.5 and 1s^{-1} were approximately 41%, 38% and 37% respectively. Whereas at strain of 0.4, the recrystallisation occurred with percentages less than that occurred at strain of 0.6, where the percentages at strain rates of 0.1, 0.5 and 1S^{-1} were approximately 29%, 26% and 24% respectively.

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