

## Abstract

The ferrite grain size of a 22MnVNb6 microalloyed steel can be estimated by developing a relationship between ferrite grain size, austenitising temperature and cooling rate from austenitising time, temperature. An extended Hall-Petch relationship was used to estimate the yield stress from the estimation ferrite grain size. Heat treatment as the annealing was used to improve the tensile and hardness properties of the steel. It was shown that the best combination of tensile and hardness properties were achieved when a higher austenitising temperature was used. Transmission electron and optical microscopy were used to study the morphology of ferrite and pearlite formed by the heat treatments. The microstructural studies showed that partition of grain by as-cast state was probably the reason for austenite and ferrite grain size improvement. Considering the experimental results, maximum errors of 14.5% and 7.5% were found in the estimation of ferrite grain size and tensile strength, respectively.

## Keywords

grain size, mechanical properties, Microalloyed Steel, Hall-Petch relationship

## 1 Introduction

The name “Microalloyed Steels” was first applied to a class of higher strength low carbon containing small additions of niobium and/ or vanadium. Any attempt at a rational definition of microalloying based on the increases in strength produced by small additions would now include aluminium, vanadium, titanium and of course niobium-treated steels. The effects of the microalloying elements are also strongly influenced by thermal treatments [1, 2]. The transformation product grain size control is an important parameter in thermal and mechanical processing of microalloyed steels. It has been well recognized that the tensile and hardness properties of steels are strongly related to the microstructure, specially the grain size [3, 4]. In the heat treatment of microalloyed low carbon cast steels involving the austenitising and cooling treatments, the parameters such as cooling rate and austenitising temperature and time have considerable influence on the ferrite grain size and tensile and hardness properties of the steels. It has been shown that accelerated cooling treatment refines the ferrite grain size while increasing the austenitising time and temperature increases the ferrite grain size. Several studies on accelerated cooling have been carried out, considering the relation of processing parameters and tensile and hardness properties to the microstructures [5]. In those studies the relationships presented for estimating ferrite grain size were deduced experimentally to relate the ferrite grain size to the cooling rate and prior austenite grain size [6-9]. The grain size estimation model presented by Umamoto et al., is extended in this research to develop a relationship between ferrite grain size, cooling rate, austenitising time, and temperature. The following Hall-Petch relationship is utilized to relate the yield stress to the ferrite grain size [10-14]

$$\sigma_y = \sigma_0 + Kd_\alpha^{-0.5} \quad (1)$$

where  $\sigma_y$  is the yield stress,  $\sigma_0$  and  $K$  are constants,  $d_\alpha$  the ferrite grain size. The grain size of a microalloyed steel has an important effect on its properties. This phenomenon is known in the literature as the grain size effect. As far as the tensile and hardness properties of the steel is concerned (such as yield and flow stress, ductility, hardness and fatigue limit), a refinement

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of the grain size generally results in an improvement of these properties at low temperature. Since the grain boundaries are known to block the movement of crystal dislocations, the grain size effect has been explained in term of the distance a dislocation can slip. The improved yield strength can be attained only by carefully controlled heat treatment. Normalizing and/or solution annealing come under consideration. Yield strength of microalloyed metallic material obeys extended Hall-Petch relationship [15, 16] as follows

$$R_y = R_o + \Delta R_a + \Delta R_g + \Delta R_p + \Delta R_t \quad (2)$$

Where  $R_o$  is friction stress,  $\Delta R_a$  is related to solid solution strengthening being linearly proportional to the concentration of alloying elements,  $\Delta R_g$  is a grain size contribution,  $\Delta R_p$  is precipitation strengthening and is the proportional to precipitation potential,  $\Delta R_t$  is matrix phase transformation strength.

### 1.1 Determination of austenite grain size distribution

Austenite grain size distribution can be recorded by photographing the microscopic picture and the determination requires measurement of each individual grain. Austenite grain size distribution in one dimension is the distribution of the lengths of linear intercepts through the grains. Metallographic techniques for measuring austenite grain size were originally base on point counting or linear intersection lengths (chords) distributions, the derivation by Spektor appears to be the first [17, 18]. The working formula is obtained [12] as below

$$N_v(j) = \frac{4}{\pi \Delta^2} \left[ \frac{n_L(j)}{2j-1} - \frac{n_L(j+1)}{2j+1} \right] \quad (3)$$

Where  $N_v(j)$  is the number of particles of mean diameter per unit volume in the interval  $2j-1$ ,  $n_L(j+1)$  is the number of chords per unit length of test line. The austenite grain size microstructures of entire the relevant samples representative photomicrographs were taken. In order to determine the austenite grain size, the samples were specially etched in a saturated picric acid solution at about 80°C [19, 20]. The austenite grain size was then measured at magnification 25× using a filler eyepiece. The degree of magnification will be limited by the fact that the picture must include a sufficient number of grain [21-23].

### 1.2 Estimation of Ferrite Grain Size

When ferrite nucleation occurs homogeneously in the austenite matrix, ferrite grain is independent of austenite grain size. In the case that ferrite nucleation occurs at austenite grain corners, ferrite grain size is proportional to  $d'_v$ . The ferrite grain size is determined by the number of ferrite nucleations at austenite grain surfaces until these surfaces are completely occupied by ferrite grains. During cooling, ferrite grains are nucleated and grown at each temperature with corresponding nucleation and growth rates. On the temperature  $T_i$  ferrite nucleates at the rate

$I_s(T_i)$  and grows at the rate  $\alpha(T_i)$ , where  $I_s(T_i)$  and  $\alpha(T_i)$  are the nucleation rate and the parabolic rate constant in the isothermal transformation at temperature  $T_i$  [24]. Ferrite grains number  $n_a$  nucleated at temperature  $T_a$  during cooling was calculated by Umemoto et al. [25] as

$$n_a = I_s(T_a) dt_a = \left[ \frac{I_s(T_a)}{Q(T_a)} \right] dT \quad (4)$$

where  $Q(T_a) = -dT/dt$  is the cooling rate. Assuming that, ferrite nucleation occurs at  $n_a$  is proportional to  $CR^n$ , where  $CR$  and  $n_1$  are the cooling rate. If ferrite nucleation occurs at austenite grain edges & corners,  $n_a$  is proportional to  $CR^{n_2}$  and  $CR^{n_3}$ . Ferrite grain size can be calculated as follows [25, 26]

$$d_\alpha = \left( \frac{2}{3S_{gb}n_\alpha} \right)^{\frac{1}{3}} \quad (5)$$

where  $S_{gb}$  is the austenite grain surface area per unit volume.

As increasing the austenite grain size and decreasing the cooling rate, ferrite grain size is increased. Determining the prior austenite grain size in heat treatment of microalloyed steels, one may substitute by the austenitising time  $t$  and temperature  $T$  as below [7, 27]

$$d_y = k_1 t^{k_2} \exp\left(\frac{-Q}{RT}\right) \quad (6)$$

where positive constants are  $k_1$ ,  $k_2$ , &  $Q$ . Therefore, the ferrite grain size is derived from Eq. (10) and (9) as follows

$$d_\alpha = a \exp\left(\frac{-A}{T}\right) (CR)^{-n} t^m \quad (7)$$

where positive constants are  $a$ ,  $A$ ,  $n$ , &  $m$ . Using this equation and measuring the austenitising time, temperature, and cooling rate, the ferrite grain size of the 22MnVNb6 Microalloyed Steel can be estimated. The estimated ferrite grain size and the Hall-Petch relationship from Eq. (1) determine the yield strength of this steel.

## 2 Experimental Procedure

With different austenitising times, temperatures, and cooling rates from the tests of heat treatment were carried out to determine of values  $a$ ,  $A$ ,  $n$ , &  $m$  from Eq. (7). A commercial strip of 22MnVNb6 microalloyed low carbon cast steel 30.00 mm long, with a rectangular cross section, 20.00 mm wide by 5.00 mm thick was applied on the tests [28-31]. The research material having chemical composition showed in Table 1. Surface processing operations for property enhancing to determine the  $d_\alpha$ , the test of heat treatment programmes are showed in Table 2 and have been utilized. Using thermocouple to determine the cooling rate of each specimen, it was utilized to record the data of temperature at austenitising time 1 s. Transmission electron

**Table 1** Chemical composition of a 22MnVNb6 Microalloyed Steel in As-Cast State used in this research work, wt-%

C	Mn	Si	P	S	Cu	Ni	V	Ti	Nb	Al	N
0.22	1.39	0.38	0.017	0.017	0.22	0.16	0.09	0.01	0.05	0.079	0.017

**Table 2** The test of heat treatment programmes used in this research study (using Eq. (8) to determine the  $d_a$ )

Austenitising temperature, °C	Austenitising time, (s)	Cooling rate, °C/s	$d_a$ (µm)
900	660	60,40,12,6.3,5	15.8,17.0,21.4,24.2,25.3
	840	60,40,12,6.3,5	17.2,18.6,23.4,26.4,27.6
	1020	60,40,12,6.3,5	18.5,20.0,25.1,28.4,29.7
	1230	60,40,12,6.3,5	19.8,21.4,26.9,30.4,31.8
1000	680	60,35,15,9,7	22.7,25.2,29.6,32.6,34.2
	860	60,35,15,9,7	24.8,27.4,32.2,35.5,37.3
	1040	60,35,15,9,7	26.6,29.4,34.6,38.1,40.0
	1220	60,35,15,9,7	28.2,31.2,36.7,40.4,42.4

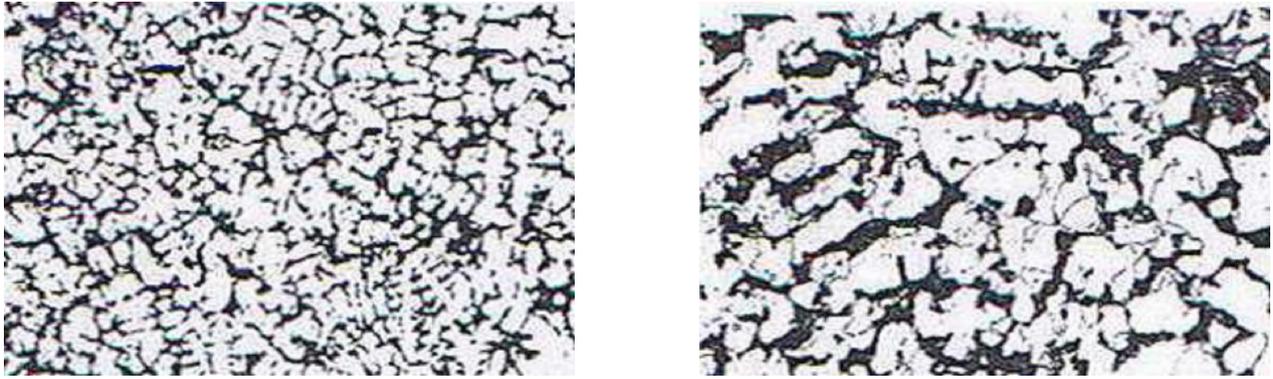
**Table 3** Size distribution of austenite grain of microalloyed steel in as-cast state

Steel	Range of chord lengths, µm	Number of chords per mm, $N_L(f)$	Diameter of grains, mm, $d_j$	Number of grains per mm <sup>3</sup> , $N_v(j)$	Evaluated mean grain size, µm, $\bar{d}$
22MnVNb6	0-170	231	0.017-0.170	$1.5417 \times 10^5$	36.1

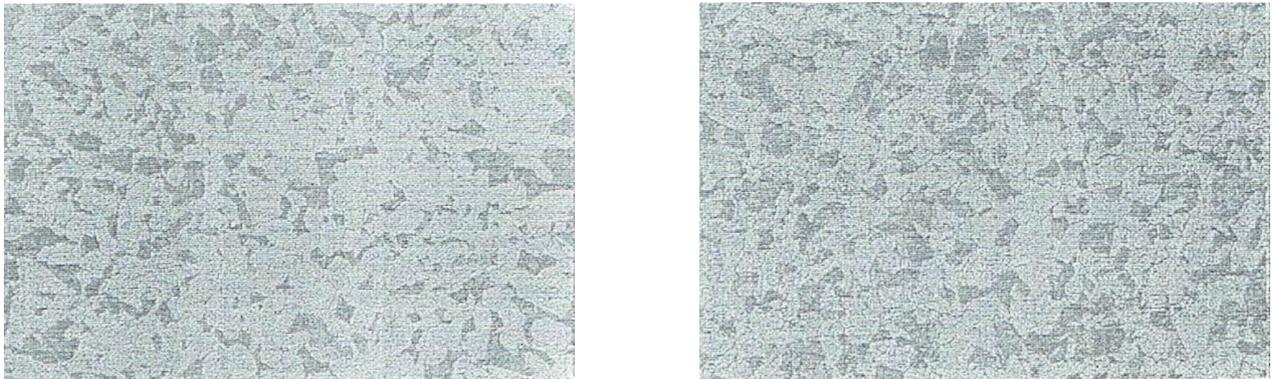
microscopy (TEM) was the most widely used technique for studying all aspects of phase transformations in steels over the length scale range 1–100 nm [32]. TEM data was used as a starting point for the proposed model in micromagnetic simulation for electrodeposited nickel nanowires [33]. Defects created by the superelastic cycling in thin Ni–Ti wires were analyzed by TEM [34]. TEM studies were carried out on the some samples of the as-cast state and the heat treatment test. Optical microscopy was used to examine the microstructure of the microalloyed steel specimen in as-cast state and the heat treatment test. In order to determine the microstructure, the samples are specially etched in 5% nital solution [9]. The concentration of the nital solution has to be adjusted for different samples. The microstructure is then measured at magnification 50x using a filler eyepiece. It was observed that the microstructures consisted of ferrite and a small amount of pearlite. The classic data in the literature for the grain size dependence of the strength in many metals are reviewed [13]. The ferrite microstructure determination should be done in a magnification suited to the size of the grain so that small grains may not be lost. The degree of magnification will be limited by the fact that the picture must include sufficient ferrite grain size. The ferrite grain size of each specimen was measured according to ISO 643 standard. To determine yield strength of the as-cast state steel of a 22MnVNb6 microalloyed, the different ferrite grain size microstructures of the steel were determined and calculated by using the Spektor equation and extended Hall-Petch relationship. After calculating  $a$ ,  $A$ ,  $n$ , and  $m$  in Eq. (7) and the extended Hall-Petch relationships for the low carbon microalloyed steel, some heat treatment as the annealing was carried out in order to compare estimated ferrite grain size and strength data.

### 3 Result and Discussion

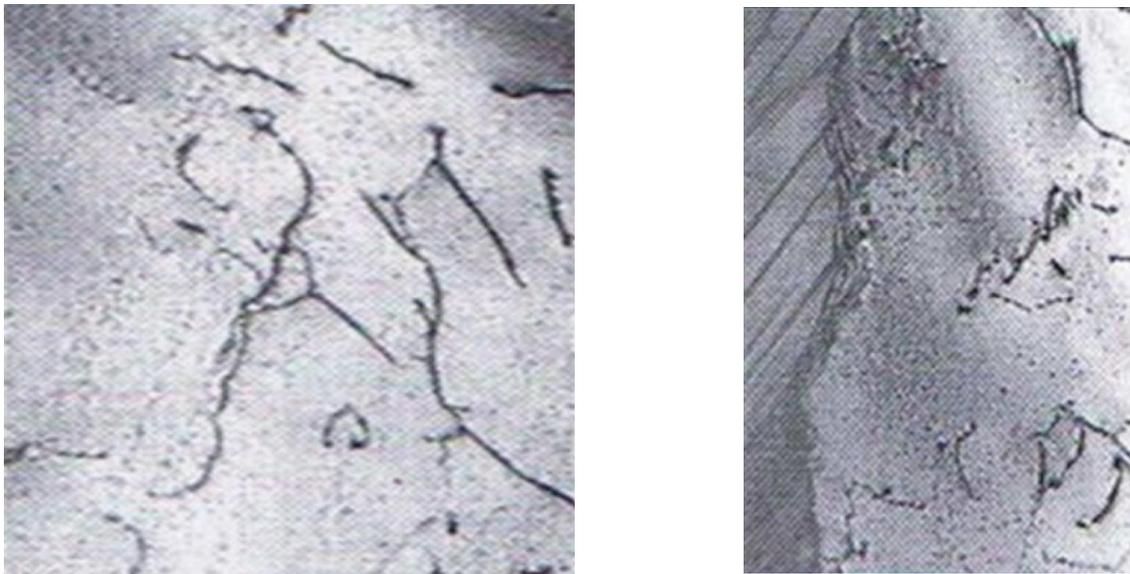
The annealing was already used to improve the tensile and hardness properties of the 22MnVNb6 microalloyed low carbon cast steel in as-cast state. Since the austenite and ferrite grain size of microalloyed steel are important factor, they were necessary to show the grain size with austenite and ferrite as in Fig. 1 and ferrite-pearlite micrograph of heat treated as annealing in Fig. 2. The microstructures were examined using light microscopy and by transmission electron microscopy of thin foils. Representative TEM images of a 22MnVNb6 microalloyed steel are given on as-cast state in Fig. 3 and on the austenitising temperature at 1000 °C in Fig. 4 [35, 36]. The austenite grain size of the as-cast microalloyed steels were investigated using Spektor's analysis in order to determine the size distribution. The prior austenite grain boundaries were revealed by etching the specimens for evaluated the mean grain size. Table 3 shows size distribution of austenite grain and Table 4, there is comparison between the mean austenite grain size of Spektor's method and the values of  $G$ ,  $\bar{d}$  &  $l$  of International standard [10, 37-40]. The microalloyed steel was studied investigated to optimize the influence of single and multiple microalloying additions on the ferrite grain size microstructure in as-cast state. Ferrite grain size distributions can be recorded by photographing the microscopic picture and the determination requires measurement of each individual grain. Ferrite grain size distribution in one dimension is the distribution of the lengths of linear intercepts through the grains. Metallographic techniques for measuring ferrite grain size were originally based on point counting or linear intersection length distribution. The ferrite grain size of microalloyed steel is important factors, on Table 5 shows size distribution and grain size contribution to yield stress. Table 6 shows estimation of



**Fig. 1** On the left, austenite grain size microstructure of the steel 22MnVNb6 in as-cast state, as 100 μm , on the right, ferrite grain size microstructure of the same steel as 100 μm , using 2 magnifications according to show some importance of pearlite



**Fig. 2** The 100 μm () ferrite-pearlite micrograph of heat treated as annealing samples of steel 22MnVNb6, 900°C (left), and 1000°C (right)



**Fig. 3** On the TEM, foil, BF as 0.2 μm , the left as ferrite microstructure of steel 22MnVNb6 and the right as interface between ferrite and pearlite of the same steel

**Table 4** Comparison of austenite mean grain size determined by Spektor's method with estimated austenite grain characteristics according to International standard (ISO643)

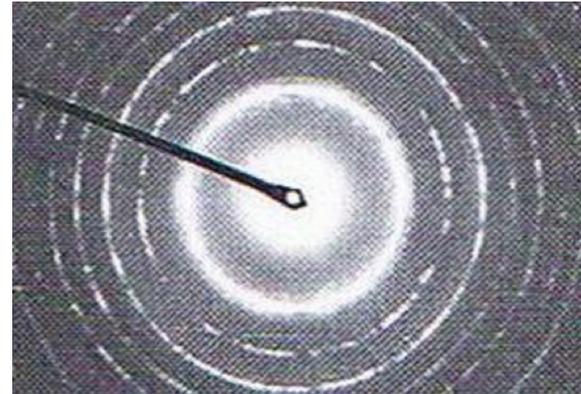
Steel	Evaluated mean grain size, μm, d	Estimated grain size (Index), G	Mean diameter of grain, μm, d	Mean intersected segment, μm, l
22MnVNb6	36.1	6.5	37.7	34.2

**Table 5** Size distribution of ferrite grains and grain size contribution to yield stress of microalloyed steel in as-cast state

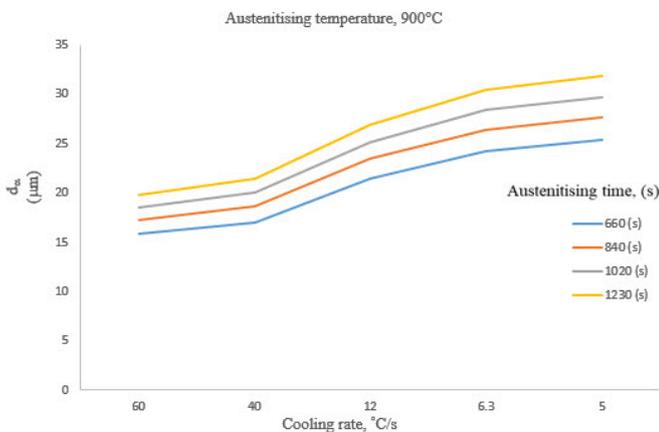
Steel	Range of chord lengths, $\mu\text{m}$	Number of chords per mm, $N_L(j)$	Diameter of grains, mm, $d_j$	Number of grains per $\text{mm}^3$ , $N_v(j)$	Evaluated mean grain size, $\mu\text{m}, \bar{d}$	$Kd_\alpha^{-0.5}$ (MPa)
22MnVNb6	0-130	625	0.013 -0.130	$14.0886 \times 10^5$	20.2	140.8

**Table 6** Estimation of yield strength of microalloyed steel on the test of heat treatment programmes, based on extended Hall-Petch relationship, with comparative mechanical properties addition

Microalloyed Steel	Heat Temp. ( $^\circ\text{C}$ )	$R_o$	$\Delta R_\alpha$	$\Delta R_g$	$\Delta R_p$	$\Delta R_t$	$R_y$ (MPa)	Vickers Hardness ( $\text{HV}_{10}$ )	Tensile Strength ( $\sigma_u$ ; MPa)
22MnVNb6	900	40	121	169	21	73	424	186	639
	1000	40	121	143	38	98	440	187	651



**Fig. 4** (Left) According to the X 20 k precipitates of ferrite grain size of the steel 22MnVNb6, (right) using the diffraction pattern TEM replica, SAD, for austenitising temperature at the 1000  $^\circ\text{C}$  to find those precipitates of the steel



**Fig. 5** Graph of ferrite grain size on cooling rate using austenitising temperature 900 $^\circ\text{C}$ , as shown orange graph of austenitising time 1230 (s), blue graph of 1020 (s), red graph of 840 (s), and azure graph of 660 (s)

$$d_\alpha = 18152 \exp\left(\frac{-10174}{T}\right) (CR)^{-0.19} t^{0.37} \quad (8)$$

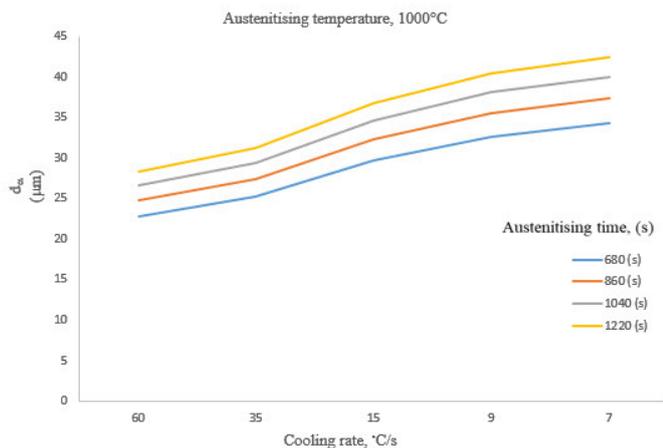
Where  $d_\alpha$ ,  $CR$ ,  $t$ , and  $T$  are expressed in  $\mu\text{m}$ ,  $^\circ\text{C}/\text{s}$ ,  $\text{s}$ , and  $K$ , respectively.

Considering the experimental results showed in Fig. 4 and 5 and Eq. (8), by increasing the austenitising time and temperature and decreasing the cooling rate, the ferrite grain size transforming from austenite is increased. In fact, by increasing the austenitising time and temperature, the prior austenite grain size is increased, and nucleation sites for ferrite formation are decreased. Atom possessing an energy level of the reactants and activation energy will have sufficient energy to react spontaneously to reach the reacted state of energy of the products [41]. A basic procedure for determining the activation energy was developed by Conrad and Wiedersich [42]. By decreasing the cooling rate, the activation energy for nucleation is decreased and a smaller number of ferrite nucleation sites produce large ferrite grain size.

#### 4 Conclusions

The Hall-Petch relationship can be utilized to examine the dependence of yield stress on ferrite grain size of the as-cast state steel, but extended Hall-Petch relationship can be utilized of the betterment that on the test of heat treatment programmes. When increased by the austenitising temperature, the effects

yield strength of microalloyed steel on the test of heat treatment programmes, based on extended Hall-Petch relationship, with comparative mechanical properties addition. To determine the  $\alpha$ ,  $A$ ,  $n$ , and  $m$  values in Eq. (8) for the test microalloyed steel, graphs of  $d_\alpha$  versus  $t$  for different austenitising temperature and cooling rates were plotted, as presented in Fig. 4 and 5. It is interesting to note that, as these figures show, the experimental relationships between  $d_\alpha$ ,  $t$ , and  $CR$  are linear. This is accordance with Eq. (8). Referring to graphs of  $d_\alpha$  versus  $t$  the ferrite grain size can be expressed as



**Fig. 6** Graph of ferrite grain size on cooling rate using austenitising temperature 1000°C, as shown orange graph of austenitising time 1220 (s), blue graph of 1040 (s), red graph of 860 (s), and azure graph of 680 (s)

of austenitising time and cooling rate on ferrite grain size are decreased. When  $d_{\alpha}$  formed by continuous cooling transformation, is decreased with an increase in cooling rate or decrease in austenitising time and temperature, the  $d_{\alpha}$  can be shown as in Eq. (8). Also, it's good correlation between experimental and estimated ferrite grain size, yield stress of the material studied of this research work.

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