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Effect of Heat Treatment on Grain Size and Mechanical Properties of 316 Austenitic Stainless Steel

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Abstract

Grain growth is one of the three important stages take place during annealing of cold-worked materials after the process of recovery and recrystallization. This paper presents the experimental analysis of the grain growth kinetics and hardness of 316 austenitic stainless steel having grain size between 6µm and 69µm. The experiments were carried out for three different temperatures and represent microscope images of grain growth during isothermal annealing at (900°C, 1000°C and 1100°C) within 30, 60, 90 and 120 min respectively. The change of grain size as a function of annealing time for various temperatures have been presented in this study the value of the coefficient n for normal grain growth was determined and found to be in the range from 0.29 to 0.37. The investigation also included the determination of the activation energy for grain growth which found to be 104 KJ/mol. The hardness of 316 austenitic stainless steel has been measured at different grain sizes and the results indicated that the hardness decreased with increasing grain size and the data in general confirm the Hall-Petch equation.

Keywords: *Grain growth, Hardness, Austenitic stainless steel, Cold-worked, Microstructure.*

1. Introduction

Austenitic stainless steels are the most common and familiar types of stainless steel. They are selected for numerous applications due to their favorable combination of characteristics such as low price, moderate to good corrosion resistance, excellent ductility and toughness along with good weldability [1]. From a metallurgical point of view, austenitic stainless steels can be made soft enough (i.e., with a yield strength about 200 MPa) to be easily formed by the same tools

that work with carbon steel, but they can also be made incredibly strong by cold work, up to yield strengths of over 2000 MPa[2].The primary objective of this research is to fundamentally understand the grain growth of austenitic stainless steels and grain size effect on the mechanical properties. Normal grain growth is defined as the uniform increase in the average grain size of a polycrystalline aggregate, due to the annihilation of small grains by grain boundary migration[3]. Grain growth is one of the three important stages take place during annealing of cold worked materials including recovery, recrystallization and grain growth. When a material is plastically deformed at a temperature that is low with respect to its melting point, it is called cold worked. Recovery is the change back of material to a stable and a lower energy condition. The mechanical properties changed due to the cold working, tend to recover their original values with slight or no change in the microstructure, this process takes place at relatively low temperature. During this stage the density of dislocations, number of vacancies and the internal stress are reduced. In addition, hardness and strength are usually somewhat reduced[4]. In recrystallization stage the cold worked grains are replaced by new ones and this usually occurs at a temperature higher than that required for recovery. The new grains nucleate at grain boundaries and increase in size until they impinge upon the neighboring grains. When the second stage of annealing is finished, new grains are formed and as the time passes at constant temperature, these grains start increasing in size. Consequently, the grain boundaries are reduced and the total surface energy is lowered, so the grain growth process occurs spontaneously due to the tendency of the system to reduce its internal energy.

2. Experimental Procedure

2.1 Materials

A rod of austenitic stainless steel with a chemical composition given in Table1 after 30% reduction in cold working with diameter of eight millimeters and six millimeters height. The specimens were cut by means of cut off machine equipped with an abrasive wheel, during cutting the specimens are cooled by water as coolant to prevent the heating of specimen due to friction, which may change the microstructure at the surface.

Table 1.Chemical composition of the 316 austenitic stainless steel

Element (Weight %)									
C	Si	Mn	P	S	Cr	Ni	Mo	N	Fe
0.02	0.84	1.6	0.01	0.015	17.8	13.3	2.22	0.001	Balance

2.2 Heat Treatment

The specimens were categorized into five groups where each group divided into four specimens. The five groups were annealed at different temperatures (700,800,900,1000 and 1100o C) for different period of time (30, 60, 90 and 120 min),

thereafter, each specimen was air cooled to the room temperature. All specimens were labeled in order to be distinguished from each other.

2.3 Specimens Preparation

The specimens were prepared for hardness and microhardness testing using metallographic laboratory . The specimens were ground on a belt sander with using water to avoid the heating that may effect on the properties and microstructure of the samples. This process was to remove all the scratches that may occurred during cutting. Due to the small size of specimens, mounting was carried out in the plastic mold, so the polishing process can be carried out more easily and faster. In the polishing silicon carbide was used as abrasive paper with 300, 400, and 600 mesh, respectively. The specimens were washed with running water and swabbed with cotton to remove adhering abrasive, then dried in the blast of warm air and stored in the desiccator. Etching carried out using a solution consists of 30 ml H2SO4, 100 ml HCl, 100 ml NHO3, and 100 ml H2O.

2.4 Hardness and Microhardness Measurements

The hardness and microhardness testing was carried out for each specimen, Vickers hardness instrument uses square based diamond pyramid with an angle of 136O between opposite faces. Microhardness test uses the same indenter but with a small test loads ranging from one to one thousand grams. The Vickers hardness load was 5Kg, while 200grs for microhardness test, the length of diagonal for square indentation was measured using microscope. The Vickers hardness number (VHN) is the ratio of the load applied to the indenter to the surface area of the indentation and can be calculated by this formula:

$$VHN = \frac{2F \sin\left(\frac{360}{2}\right)}{L^2} = \frac{1.854\ F}{L^2} \quad (1)$$

Where:

F = the applied load, Kg
L= the mean diagonal of the indentation, mm

2.5 Grain Size Measurement

Measurements of various grain geometry parameters, grain area (A), equivalent diameter (deq), maximum dimension (dmax), grain perimeter (p),and calculation of αand β were performed from micrographs taken of two randomly selected fields from each specimen, the shape factor α = dmax/deq and β = p/deq. The grain area (A) and the equivalent diameter (deq) can be directly used to characterize the grain size of individual aggregates. On the other hand α and β describe the shape of individual aggregates, with α being sensitive to grain elongation (α = 1 for a circle) and β being sensitive to the curvature of grain boundary. All the measurements were analyzed in terms of mean value of area, E(A), mean value of grain equivalent diameter E(d) and coefficients of a variation, CV(A) and CV(d).

$$CV(X) = \frac{SD(X)}{E(X)} \quad (2)$$

Where: SD(X) is standard deviation.

3 Results and Discussion

3.1 Grain Growth

The grain size as a function of the annealing time with various temperatures is shown in Table 3 and the results are presented in Figures 1,2 and 3 that show the different of grains growth during isothermal annealing at 900oC, 1000oC and 1100oC, respectively. In an effort to identify the beginning of recrystallization, annealing was done at low period of time (5,10 and 15min) and no recrystallization was observed. When annealing was carried out at 900oC for 30 min the recrystallized structure was observed. Figures 1,2 and 3 demonstrate the effects of holding time on grain growth at different temperatures, as it is clear, the average grain size increases with

increasing holding time from 30 min to 120 min, as well as, at annealing temperature increases the boundaries between annealed grains migrate and larger grain grow by an increase in the average grain size. The experimental results are plotted in Figure 4 on logarithmic coordinates. The points which have obtained experimentally in this work fall reasonably well on nearly parallel straight lines, the results confirm relatively closely to the following known empirical equation (the equation of grain growth) [5]:

$$D = \mathrm{K}\, t^{n} \qquad (3)$$

Where D is the average grain size, K is a constant of proportionality that relates heating temperature and activation energy for grain growth, and t is the total time of annealing, which is the sum of the time necessary for complete recrystallization and that of the grain growth[6].The values of n were determined from the slopes of the straight lines plotted in Figure4, which found to be ranged from 0.29 to 0.37.In Table 4 and Figure5 that show the logarithm of the slopes of these parallel lines as a function of the reciprocal of the temperature (log D2/t plotted versus1/T). The data give straight lines from which estimated that the activation energy Q for grain growth in 316 austenitic stainless steel is about 104 KJ/mole.

There is a difference between the n-values which are obtained experimentally from one investigator to another and this depends on the accuracy in measurements as well as on the material tested. In general the n-values are smaller than 0.5 which is obtained theoretically, for a constant distribution of grain size the grain boundary velocity is proportional to the driving force for growth, and inversely proportional to the grain size [7]. During the heat treatment of austenitic stainless steel there are different precipitation reactions can take place, the type of precipitates and their place of formation is strongly dependent on the time and temperature of annealing and the amount of cold working prior to annealing[8].Since the specimens are relatively heavily deformed about 30% cold worked, so it would not be surprising to expect the formation of intermetallic phase during recrystallization at grain boundaries and triple grain junctions as well as precipitation of carbide M23C6 at grain boundaries and as the recrystallization process progresses more carbide is precipitated at these grain boundaries. In

the temperature of annealing of 9000C which is relatively low, it is unlikely that these precipitates (carbides and intermetallic phases) retard the grain growth process, unless after long time, so the driving force for grain growth is high which can lead to high rate of grain growth (i.e. high time exponent, n= 0.37).The coalescence of precipitate particles occurs in all the metallic systems at elevated temperatures which is a special significance in view of the effect of particle size on the energy barrier which can retard grain growth when coalescence has permitted the particle size to reach a critical size, so the driving force for grain growth is decreased[9]. This hypothesis is in satisfactory agreement with the present work results, where the annealing at higher temperature (10000C), the rate of grain growth was lower (i.e. the time exponent n was reduced to lower value n=0.29). So, the reduction in the value of time exponent n can be attributed to the coalescence of precipitates. At higher temperature of annealing (11000C), the time exponent was observed again increasing (n=0.37).The grain growth rate can be increased when the coalescence causes the particle size to exceed a critical value and dissolution of the precipitates is not a necessary requirement for growth to progress[9].

In the present study the time of annealing was two hours maximum, so this time was too short for dissolution of the precipitated particles to occur, but the coalescence of particles and exceeding the critical size could be a reason for that change in the rate of growth.

3.2 Hardness and Microhardness Measurements

The results for the hardness and microhardness are given in Figures6,7and Table 5. These results indicated that the hardness decreases noticeably with increasing the grain size. The relationship between the hardness and grain size can be described by the Hall-Petch equation:

$$Hv = Ho + KH\ d^{-1/2} \qquad (4)$$

Where Hv is the Vickers hardness, Ho is intercept and KH is the slope and these values can be obtained from the Figure 6 where Ho= 114 kg/mm2 and KH= 30.7 kg/mm3/2. Therefore, the Hall-Petch equation can be expressed as:

$$Hv\ (kg/mm2) = 114\ kg/mm2 + 30.7\ kg/mm3/2\ d\text{-}1/2\ (mm\text{-}1/2) \qquad (5)$$

A careful examination of the obtained experimental points suggests the existence of three linear relationships instead of one as shown in Figure 6.Each set of points contains the data for the specimens annealed at the same constant temperature which was different for each case. The dependence of hardness on grain size is numerically different in the three regions with different values of Ho and KH as indicated in Table6. The selection of three lines is based on the fact that when a single line is drawn using the entire data points, the result was shown in somewhat a poor fit and the points are more scattered. The three lines were proposed where the data points almost exactly fall on three lines. The three different annealing temperatures are likely to produce three different types of microstructure with varying content of precipitates. Such conclusion is further supported by detected differences in grain growth kinetics. The microstructure of specimens differ in the degree of grain size uniformity as measured by CV(A) and this effect should also be taken into account.

The microhardness measurements were carried out for the same material under the same conditions. According to the Hall-Petch equation the values of Ho and K are 127 kg/mm2 and 37 kg/mm3/2,respectively. The values are different from the values of hardness as expected due to the applied load and type of indenter. The measurements for the same grain size were found to be highly scattered and especially for small grain sizes. For the microhardness where low loads were used, the hardness within the grain interiors and in the vicinity of grain boundaries could be varied due to the difference between grain boundaries and grain interiors and particularly visible for fine grained material where more boundaries are present and probability that indentation can be localized at boundaries increasing. Generally, the microhardness measurements are less accurate and not reliable.

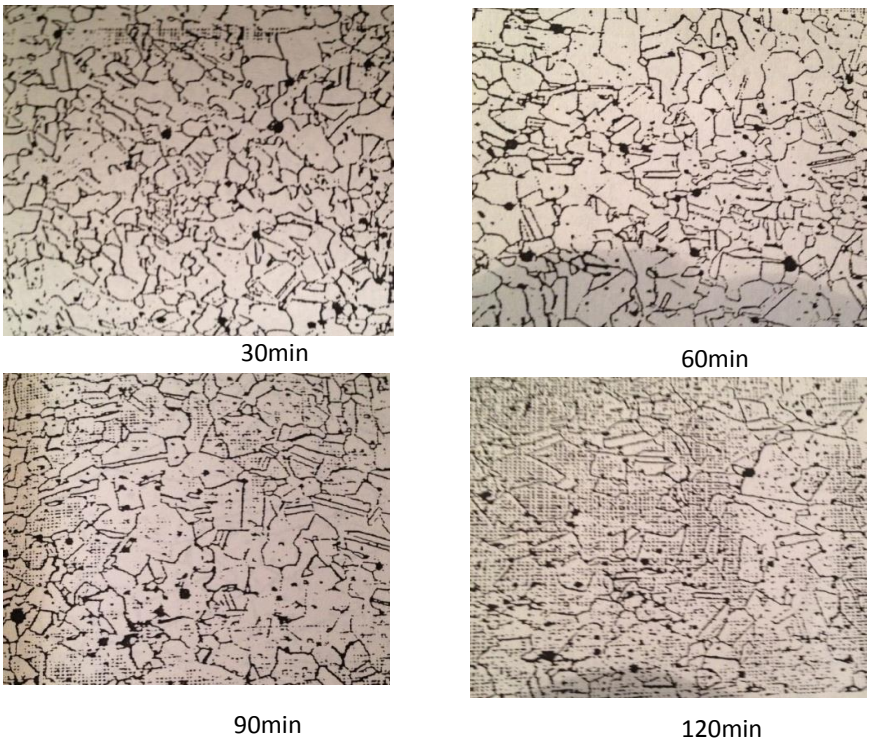
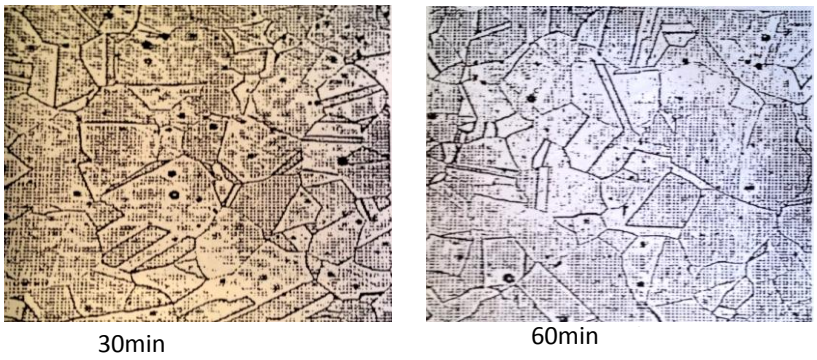
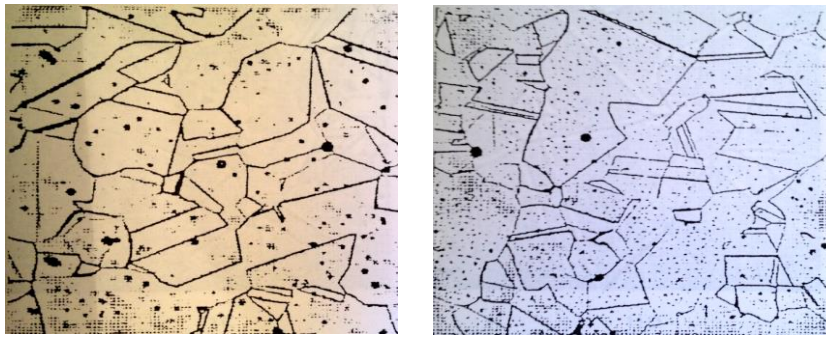


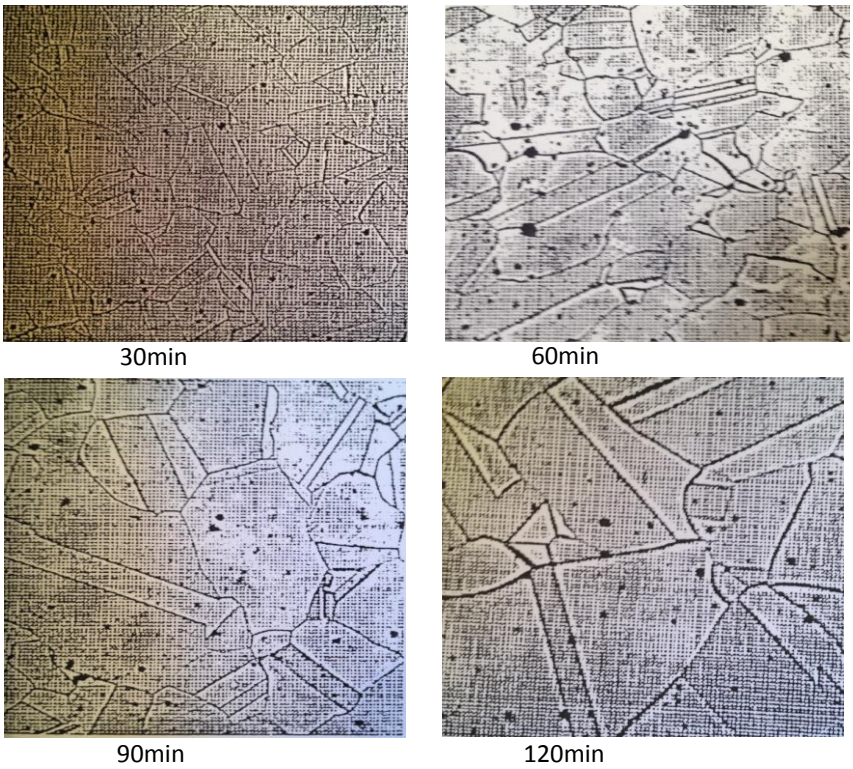
Figure 1.Variation in grain growth with time at 900°C(500X)





90min 120min

Figure 2. Variation in grain growth with time at 1000°C (500X)



30min 60min

90min 120min

Figure 3.Variation in grain growth with time at 1100°C (500X)

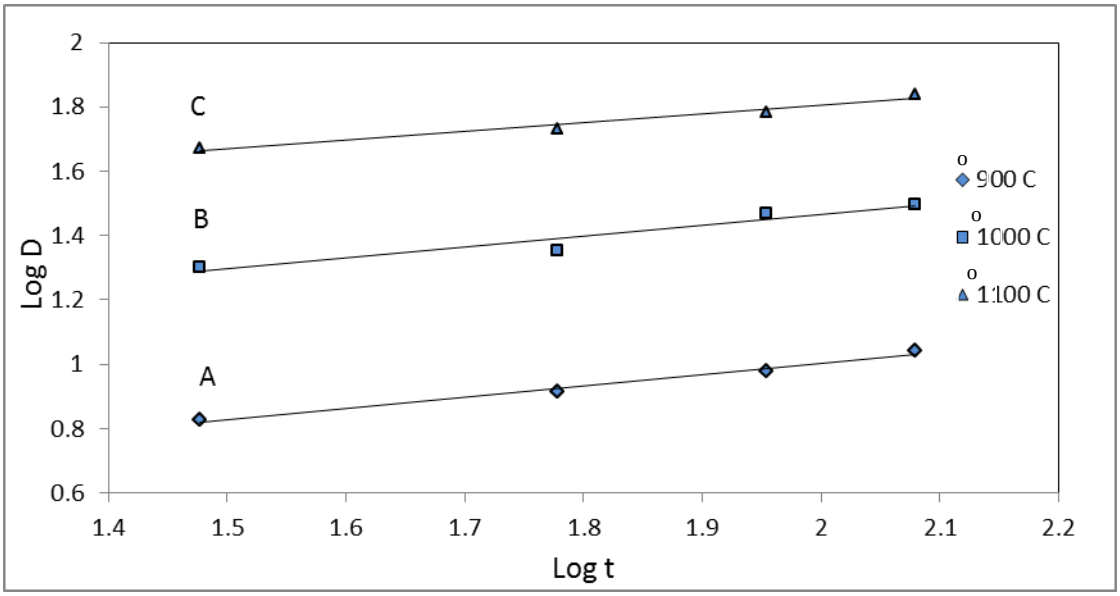


Figure 4. Grain size as a function of the annealing time and temperature

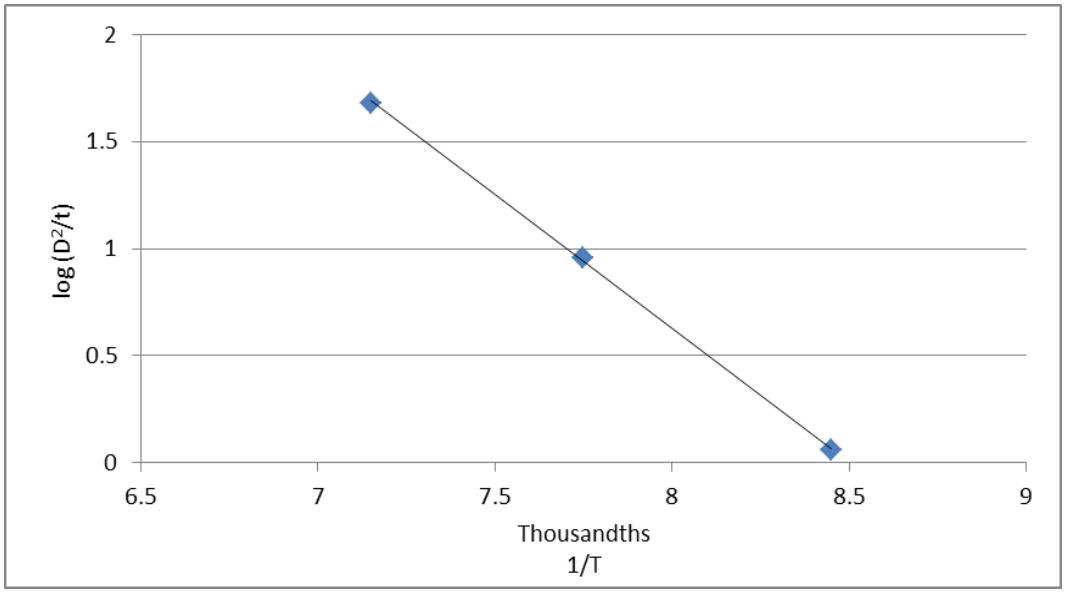


Figure 5. The logarithms of the slope of the isotherms as a function of the reciprocal of absolute temperature

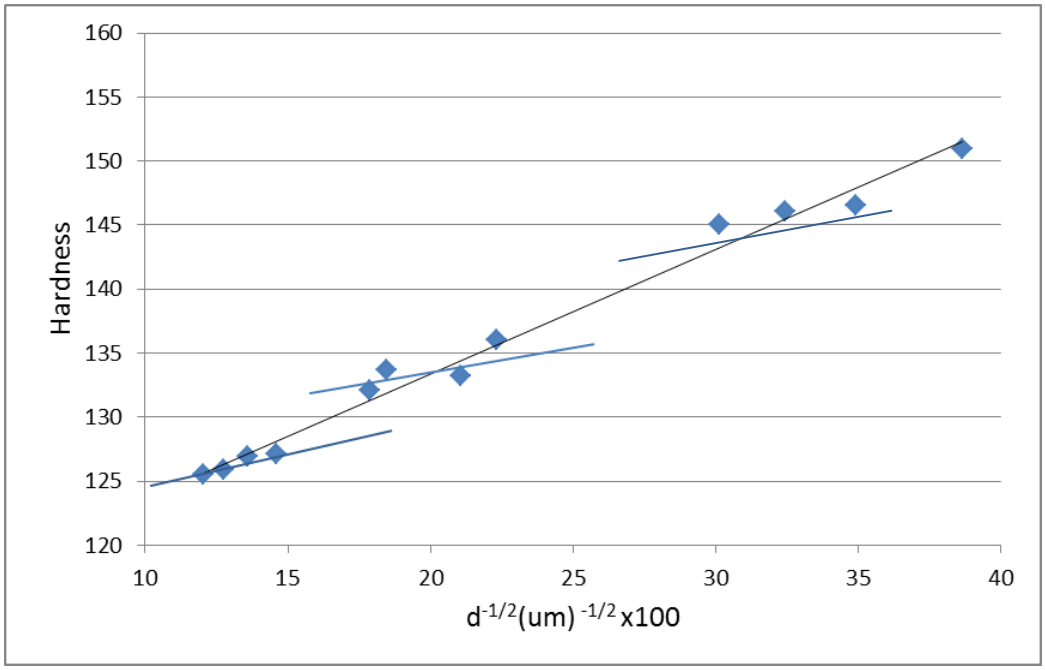


Figure 6. Variation in Vickers hardness with $d^{-1/2}$

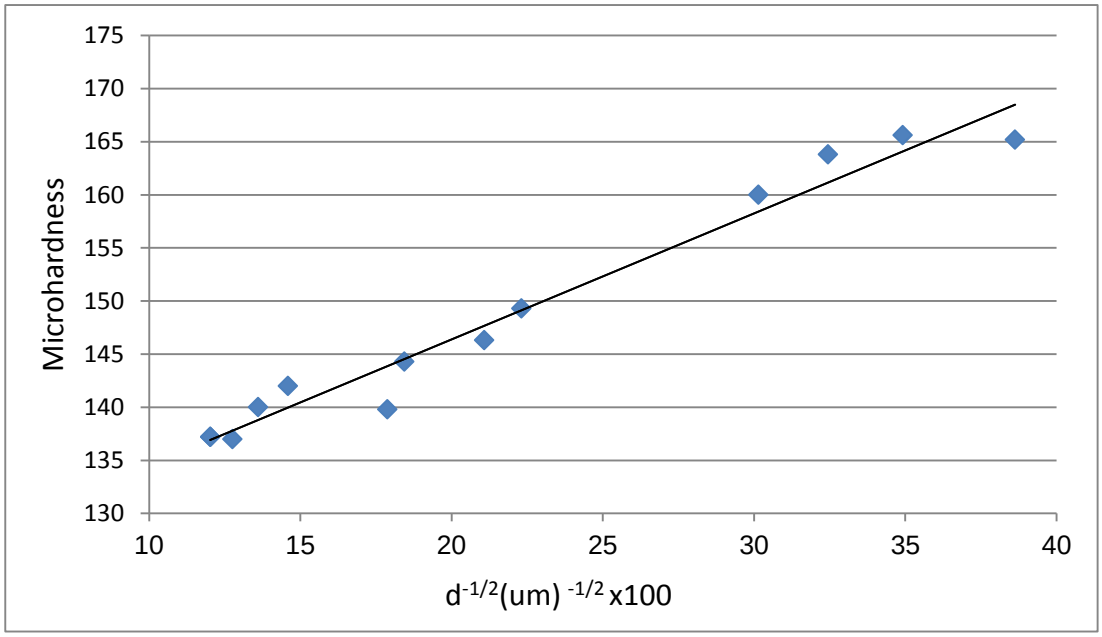


Figure 7. Variation in Vickers microhardness with $d^{-1/2}$

Table2. Measurements of various grain geometry parameters

Specimen	E(A) (μm)^2	SD(A)	CV(A)	E(d) (μm)	SD(d)	CV(d)	α	β
9A	42.6	41.066	0.964	6.7	3.015	0.450	1.354	3.807
9B	63.4	60.103	0.948	8.2	3.813	0.465	1.322	3.796
9C	84.2	77.043	0.915	9.5	4.1135	0.433	1.300	3.689
9D	113.9	106.72	0.937	11.0	4.983	0.453	1.344	3.842
10A	411.1	501.95	1.221	20.1	10.8339	0.539	1.336	4.000
10B	517.5	595.13	1.150	22.5	12.2175	0.543	1.385	4.111
10C	896.4	1061.3	1.184	29.4	16.758	0.570	1.311	3.900
10D	951.3	949.4	0.998	31.3	15.337	0.490	1.305	3.726
11A	2137	2147.7	1.005	47.0	22.607	0.481	1.317	3.802
11B	2863	2777.1	0.970	54.0	27.054	0.501	1.284	3.748
11C	3501.5	2860.7	0.817	61.5	26.0145	0.423	1.175	3.545
11D	4623.2	4549.2	0.984	69.2	33.216	0.480	1.497	4.168

Table 3. Grain size as function of annealing time and temperatures

Temperature(^{o}C)	t (min.)	D (μm)	log t	Log D
900	30	6.7	1.477	0.8260
	60	8.2	1.778	0.9138
	90	9.5	1.954	0.9777
	120	11.0	2.079	1.0414
1000	30	20.1	1.477	1.3032
	60	22.5	1.778	1.3522
	90	29.4	1.954	1.4683
	120	31.3	2.079	1.4955
1100	30	47.0	1.477	1.6720
	60	54.0	1.778	1.7324
	90	61.0	1.954	1.7850
	120	69.0	2.079	1.8388

Table 4. Change oflog(D^2/t) as a function of the reciprocal of the absolute temp.

D (μm)	D^2(μm)^2	t (min)	log(D^2/t)	avg. log(D^2/t)	T (K)	1/T (K^{-1})
6.7	44.89	30	0.1700	0.0586	1173	0.008525
8.2	67.24	60	0.0490			
9.5	90.25	90	0.0120			
11.0	121.00	120	0.0036			
20.1	404.00	30	1.1290	0.9560	1273	0.007855
22.5	506.25	60	0.9260			

29.4	864.40	90	0.8500			
31.3	979.70	120	0.9190			
47.0	2209.0	30	1.8670	1.6800	1373	0.00728
54.0	2916.0	60	1.6800			
61.0	3721.0	90	1.6160			
69.0	4761.0	120	1.5900			

Table 5. Hardness and microhardness measurements

Specimen Designation	**Hardness HV_5(Kg mm^2)**	**Microhardness HV_5(Kg mm^2)**	**Grain Size d (μm)**	**$d^{-1/2}$ (μm)$^{-1/2}$.10^2**
9A	151.0	165.2	6.7	38.633
9B	146.4	165.6	8.2	34.921
9C	146.1	163.8	9.5	32.444
9D	145.0	160.0	11.0	30.151
10A	136.0	149.3	20.1	22.305
10B	133.2	146.3	22.5	21.082
10C	133.7	144.3	29.4	18.442
10D	132.1	142.0	31.3	17.874
11A	127.1	140.0	47.0	14.586
11B	126.9	139.8	54.0	13.608
11C	125.5	137.2	61.5	12.751
11D	125.9	137.0	69.2	12.021

Table 6. Values of H_o and K_H in three different regions

Constant	**Region I**	**Region II**	**Region III**
H_o (Kg/mm^2)	119	126	128
K_H (Kg/$mm^{3/2}$)	17	12.26	17.88

4. Conclusions

- At relatively low temperature 900°C the rate of grain growth is high with time exponent in the grain growth law (n=0.37). At this temperature some carbides and intermetallic phases are nucleated, but their size is a small for a short annealing time, so the driving force is high.
- At annealing temperature of 1000°C, the rate of grain growth is reduced with time exponent n=0.29 and this is probably due to the critical size of particles, resulting from the coalescence

of precipitates (carbides and intermetallic phases)where they can retard the migration of grain boundaries and grain growth process.

- At higher temperature of annealing (1100°C) the formed precipitates exceeds the critical size and their effect on grain growth process can be neglected and therefore the rate of grain growth is accelerated with time exponent n=0.37.
- The grain growth is not significant at short times less than 15min even at high temperatures(1100^{o} C).
- Hardness in general decreases with increasing grain size.
- Careful examinations of the obtained experimental data have given three sets of points with different values of $_{Ho}$ and $_{KH}$. Each set contains the data from the specimens annealed at a constant temperature.

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