Research Note

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Recrystallization Behavior of Deep Drawing Low Carbon Steel Sheets Produced by Mobarakeh Steel Plant

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Abstract

In the present work the recrystallization behavior of deep drawing grade Al-killed low carbon steel sheets produced by Mobarakeh Steel Plant (MSP) has been studied. Salt bath isothermal annealing has been applied to cold rolled samples and the resulted microstrucrures have been compared with the microstructure obtained from the box annealing process in MSP. It is found that at 635 and 700°C the isothermal annealing produces equiaxed grains, while box annealing process presents an elongated grain structure. The recrystallization fractions have been measured using the point counting method. The linear intercept method has been employed to measure the grain size. It is revealed that at 550°C, in salt bath annealing, recrystallization is stopped after 50% recrystallization with elongated new grains. Constant grain sizes upon holding after recrystallization at 635 and 700°C during isothermal annealing show that the grain growth is stopped. Further holding at 700°C results an abnormal grain growth in the recrystalized microstructure. The hardness variations during annealing, with a decrease at the beginning of annealing followed by an increase trend at all temperatures, show a good consistency with microstructural observations. Investigation of the results leads to the conclusion that a precipitation process occurs during annealing which greatly affects both restoration processes and resulted microstructure as well as the properties. The effect of the precipitation process depends on the time of its occurrence with respect to each restoration process. A Relatively high value of 315 KJ/mole for recrystallization activation energy has been related to the simultaneous occurrence of recrystallization and precipitation processes.

Keywords: Low carbon steel, Recrystallization, Mirostructure, Gain growth, Pecipitation, Mobarakeh

Introduction

Aluminum-killed low carbon steel sheets are widely used in automotive industry [1,2)]. Their deep drawability together with ductility are the most important parameters making the sheets suitable for their applications [3,4)]. Desired properties may be obtained by appropriate thermomechanical processing on a materialwith controlled composition[5)].

Therefore, the understanding of manufacturing parameters, final microstructure, mechanical properties and their interdependent relationships play a key role in the economical production of this kind of steel sheet [6, 7)].

Thermomechanical processing including reheating temperature and holding time of slabs, hot rolling conditions and cooling rate, coiling temperature, cold rolling reduction and annealing of cold worked sheets altogether determine the final microstructure [8,9)].

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During the thermomechanical treatments different metallurgical phenomena such as strain hardening, dynamic and/or static recovery and recrystallization and grain growth may occur, which affect the final properties [3, 5, 10)]. The existence of carbon, nitrogen, aluminum and some carbide forming elements in low carbon steel may give rise to precipitation or dissolution of precipitates at different stages of the process which in turn lead to a wide range of particle size and distribution [11,12)]. These precipitates may greatly affect the metallurgical phenomena during the thermomechanical treatments. Therefore, they present an important role in the final microstructure and mechanical properties of steel sheets [13, 14)].

In the present work the restoration processes during annealing of a deep drawing quality Al-killed low carbon steel produced by MSP is studied. The object of the investigation is to understand the effect of various parameters on the microstructure as well as their influences on the mechanical properties of the steel sheets.

Materials and Methods

An aluminum- killed low carbon steel has been investigated in the present work. It was received

Table 1. Chemical composition of low carbon steel (wt%).

Element	C	Mn	P	Si	S	N	Al	Cr	Mo	V	Ti	Nb
wt%	0.04	0.21	0.007	0.011	0.005	0.004	0.05	0.01	0.001	0.004	0.002	0.004

from MSP. The average chemical composition for this steel is shown in Table 1. The material was re-heated to 1260°C, held for three hours, subsequently hot rolled to 2mm in thickness with finishing temperature of 870°C, and finally coiled at 570°C. The hot bands were cold rolled 65% reduction in area at room temperature to get 0.7mm in thickness. Then the cold rolled specimens with dimensions of 0.7×10×19 mm were prepared. To investigate the kinetics of recrystallization and the effect of annealing temperature and holding time on the recrystallization behavior, the specimens were isothermally annealed at 550, 635 and 700 °C, in a salt bath. A thermocouple was attached to each sample to monitor the temperature changes with a data logger during the annealing process. At the end of annealing time, the samples were quenched in water. The specimens were polished down mechanically to ¼ micron in longitudinal section and etched in 0.5% nital for 3-4 min to reveal both the microstructure and substructure [15]. The progression of recrystallization was followed by the point-counting method. The mean linear intercept method was used to measure the grain sizes. The observations and microanalysis were performed on the "Lica" image analyzer. The effects of annealing temperature and holding time on mechanical properties of the present low carbon steel were studied using the Vickers microhardness scale. The hardness was measured on polished longitudinal sections of cold rolled and / or annealed specimens.

The same procedure of metallography and optical microscopy were applied on specimens cut from annealed sheet produced by box annealing in MSP. The maximum temperature in the MSP box annealing process is 715°C with very much slower heating and cooling rate than in isothermal salt bath annealing.

Results

Figure 1 shows the microstructure of specimen cold rolled 65% reduction in area on longitudinal section. The grains are elongated in direction of rolling and cold worked substructures with series of microbands are observed within them. The microbands are inclined to the rolling plane and in some grains they appear in two distinct directions. Also, a number of dark particles are present within the microstructure. It seems that some broken phases appear at the original grain boundaries, which are aligned along the rolling direction.

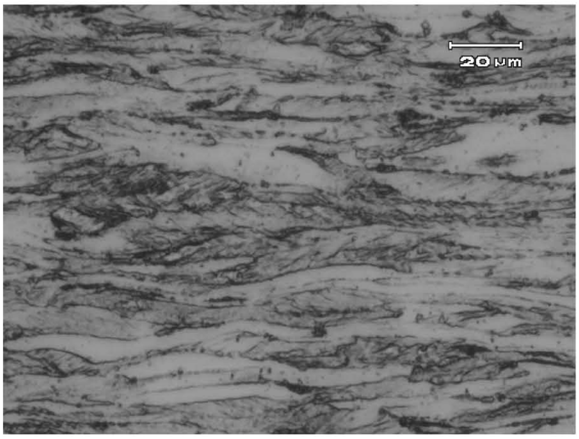

Fig. 1. The cold rolled microstructure after 65% reduction in area.

Figure 2 presents the microstructural changes during annealing at 550°C for different holding times. After 60s, the appearance of new grains at grain boundaries indicates that the recrystallization has begun. At longer holding time, the recrystallization has proceeded. Large completely recrystallized grains coexist with regions of deformed grain surrounded by small new grains indicate an inhomogeneity of recrystallization process, Figure 2b. After a long time it seems that the recrystallization process stops and the recrystallized grains appear elongated in shape resembling original cold rolled ones, Figure 2c. In some samples the recrystallization has proceeded significantly in some areas, while in the adjacent areas it has hardly started. These recrystallized and unrecrystallized areas are periodically observable, Figure 2d.

The microstructural evolution during annealing at 635°C for various holding times is illustrated in Figure 3 The progression of recrystallization process is evident in the micrographs. At the final stages, it is difficult to distinguish between the recrystallized and unrecrystallized grains. However, the substructures within some grains are considered as evidences of unrecrystallized regions.

Figure 4 shows the microstructures of specimens annealed at 700°C for different holding times. After 30s, the beginning of recrystallization is evident, Figure 4a. With increasing holding time, the overall average grain size decreases and the original deformed grains containing substructural features coexist with new small grains. The longer the holding time, the less the deformed grains will be and a wide distribution of sizes is evident, Figure 4b. The recrystallization process has completed after 9hr at this temperature. The recrystallized grains appear equiaxed in shape, Figure 4b. After long annealing time, the abnormal grain growth occurs in some regions of the microstructure and some relatively coarse grains are dispersedly observed among the small grains, Figure 4c.

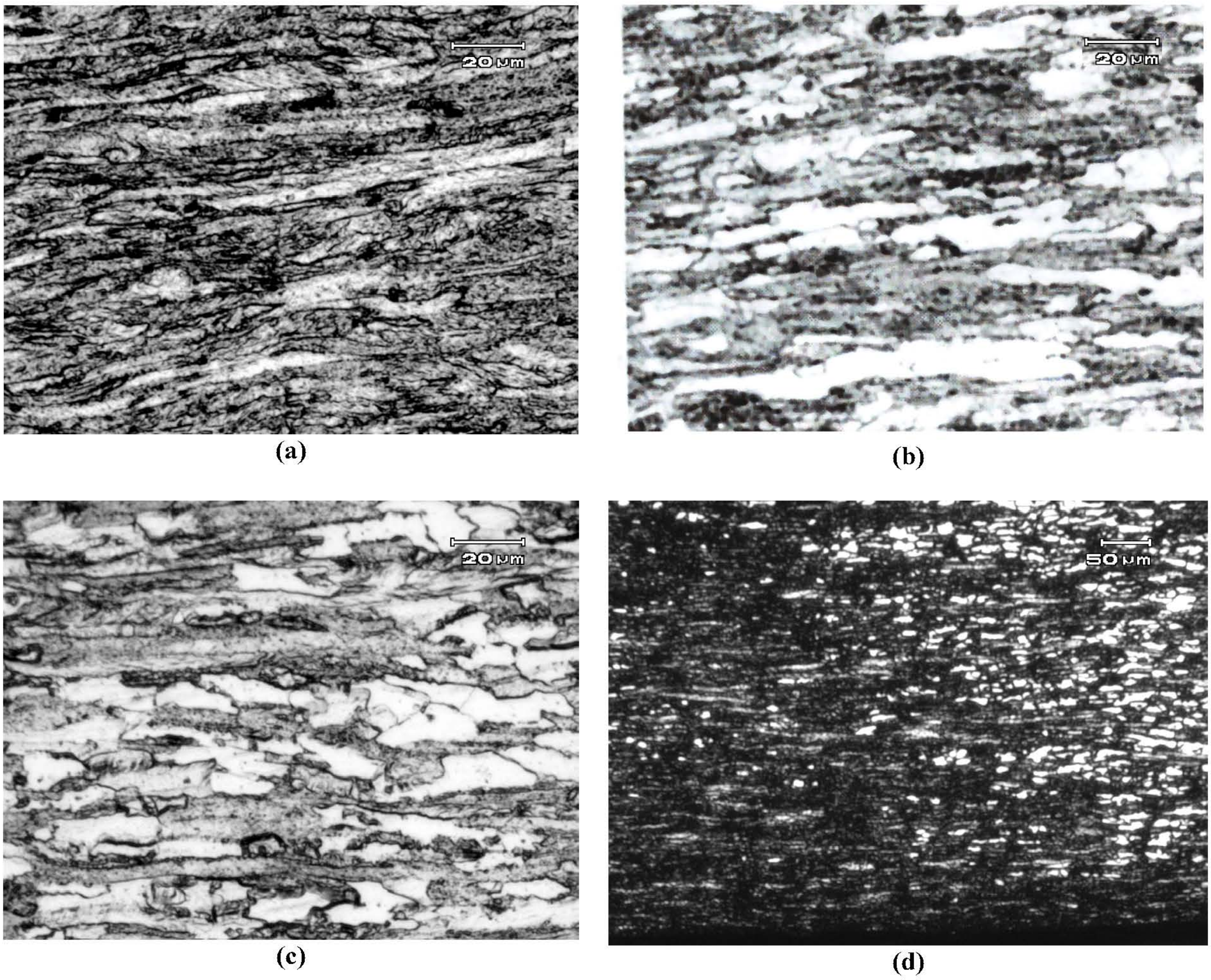

Fig. 2. Microstructural evolution during annealing at 550°C, a) 1min, b) 6 hr, c) 89 hr and d) 47hr.

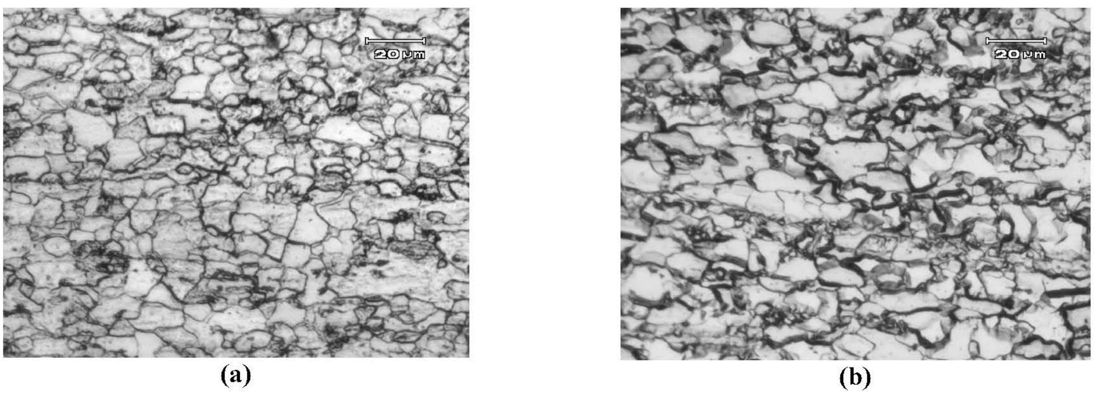

Fig. 3. Microstructural evolution during annealing at 635°C, a) 1min and b) 46hr.

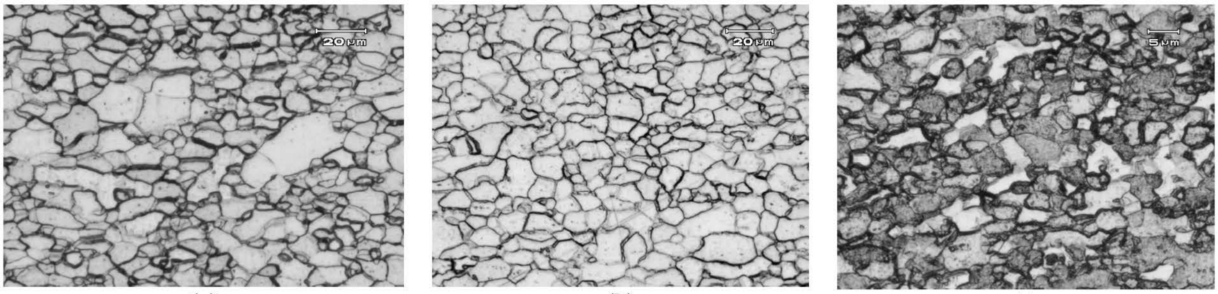

Fig. 4. Microstructural evolution during annealing at 700°C,
a) 30 sec, b) 9hr and c) 24hr.

The microstructure of sample annealed at MSP heat treatment cycle is illustrated in Figure 5. The morphology of grains is elongated and substructures are evident in some, which are the indications of uncompleted recrystallization in those areas. Compared with the microstructures of samples annealed at 700° C of the present work, the grains are coarser.

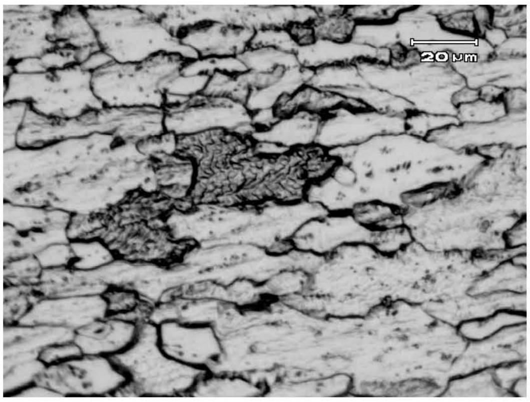

Fig. 5. Recrystallized microstructure of sample annealed in MSP box annealing process.

The results of quantitative measurements of the variation of recrystallization fraction with annealing time and temperature are illustrated in Figure 6. The curves assume so-called Avrami sigmodal shapes against log-time. As it is seen, the incubation time increases with decreasing temperature. The recrystallization process has completed at 635 and 700°C, but it has ceased after about 50% at 550°C.

The effects of annealing temperature and holding time on the grain size are illustrated in Figure 7. At both annealing temperatures, the average grain sizes initially decrease with the progression of recrystallization process. It is followed by a small increase at longer holding times. However, constant grain sizes upon longer holding times suggest that grain growth does not occur.

Figure 8 shows the effect of annealing temperature and holding time on the Vickers hardness of the material. The hardness decreases rapidly at the beginnings of the annealing process at all temperatures, but it shows an increase trend with increasing holding time. This increase is more significant at 550 and 635°C.

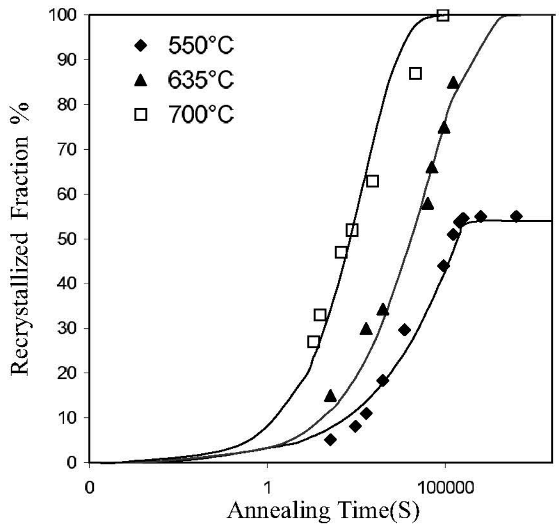

Fig. 6. Recrystallization behavior during annealing at different temperatures.

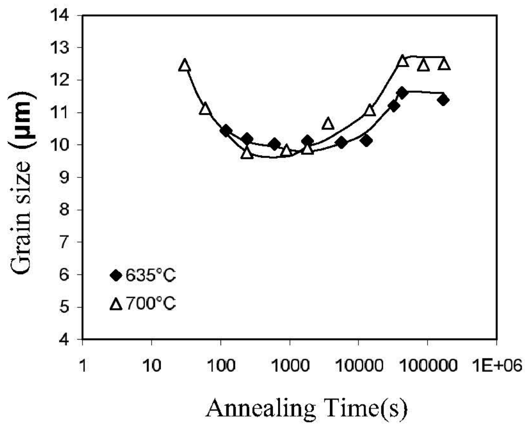

Fig. 7. Grain size variations during annealing at different temperatures.

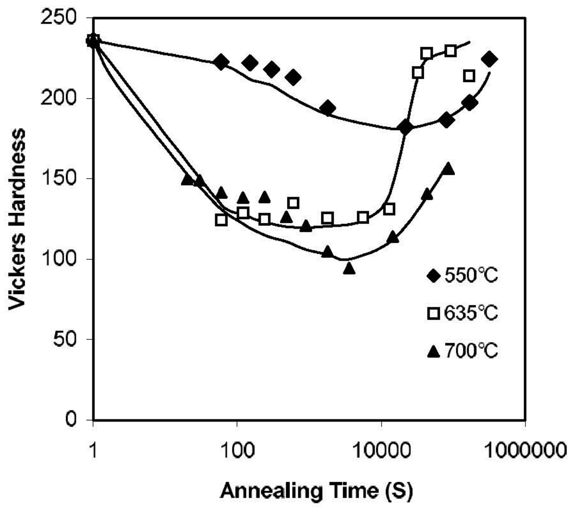

Fig. 8. Hardness variations with time at different temperatures.

Discussion

The main characteristic of cold- rolled structure is the presence of bands within the elongated grains. They have given rise to the observation of creased grains and grains containing fish bone structure, Figure 1 They are microbands and/or shear bands, which appear in all cold worked metals [16-20]. These bands are evidences of inhomogeneity in cold- rolled structure and have very significant effects on nucleation and growth stages in the recrystallization process during subsequent annealing. They may provide very potent nucleation sites for recrystallization in addition to those which may form on original boundaries in cold worked grains [10,15,21].

The quantitative measurements show that the decrease in annealing temperature leads to an increase in recrystallization start time and a decrease in recrystallization rate, Figure 6. Longer incubation time at lower temperature indicates the effect of temperature on the kinetics of the recrystallization process. After long holding times, recrystallization stops at 550°C after about 50% recrystallization. The long incubation time at this temperature and the concurrent recovery reduce the driving force for progression of recrystallization [5]. But complete recrystallization has been reported at this temperature in low carbon steel under the same conditions [22,23]. Therefore, the cessation of recrystallization in the present material should be attributed to another process, which induces a pinning effect on the boundaries of recrystallizing grains. It is interesting to note that at low temperatures, recrystallizing areas have grown inside the original elongated grains and new grains are confined within the old cold worked ones. It suggests that the original boundaries are pinned and that they inhibit the growth of new grains. So the final recrystallized structure inherits the morphology of the cold-rolled one, Figure 2d. A similar elongated morphology is evident in the recrystallized structure of samples annealed in MSP box annealing cycle, Figure 5.

At higher temperatures, 635 and 700°C, the recrystallization process proceeds to completion. The morphology of recrystallized structures shows a significant difference at low and high temperatures. At 550°C, it preserved the elongated shape of cold-rolled grains while the grains appear equiaxed in shape in the recrystallized structure at 700°C. Completion of recrystallization with equiaxed structure at higher annealing temperature indicates that recrystallization is not significantly influenced by the pinning inducing process as it was observed at 550°C.

Activation energy, Q_{rex}, for recrystallization was obtained using the following equation:

$$1/t_{50} = A \exp(-Q_{rex}/RT) \qquad (1)$$

where, t_{50} is the time for 50% recrystallization [22]. The variation of t_{50} versus reciprocal absolute annealing temperature is illustrated in Figure 9 .The apparent activation energy was calculated as 310kJ/mole. The activation energy for recrystallization for low carbon steel has been reported in the range of 125- 400 kJ/mole [24,25]. The relatively high calculated value of Q_{rex} implies that another process, probably a precipitation one, takes place during the recrystallization process in accord to the previously discussed results [24].

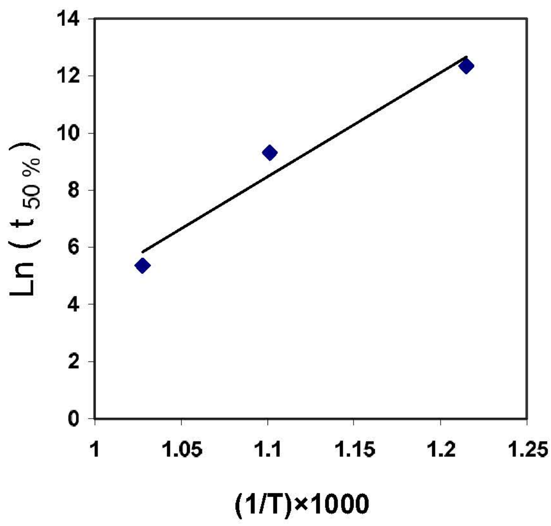

Fig. 9. Time for 50% recrystallizaiton versus reciprocal temperature.

The grain growth behavior in the present material is interesting. The nearly constant grain size at 635 and 700°C, after recrystallization, with different holding times, Figure 7, suggests that grain boundaries are pinned. At 700°C a different story takes place. After enough long holding time, very large grains surrounded by small ones, are frequently observed, Figure 4c. This phenomenon may be attributed to the unpinning of some boundaries resulted from dissolution or coarsening of fine pinning particles [22)].

Vickers hardness variation with annealing temperature and time is in agreement with above mentioned observations [8)]. At the early stages of annealing, hardness decreases at all temperatures indicating the onset of recovery, which is more significant at higher temperatures. As time goes on, the effect of annealing temperature becomes more evident. At 550°C, no significant decrease in hardness is observable while at 635 and 700°C substantial decrease in hardness resulted from recovery and recrystallization shows a good consistency with the microstructural observations. In all diagrams, Figure 8, an increase trend is observed at longer times which may be caused by a precipitation hardening process [13)].

As discussed above, recovery and recrystallization processes are intervened by another process which is most likely a precipitation process. The present material is an Al-killed low carbon steel, which has been reheated to 1260°C and held for three hours before hot rolling stage. Therefore, aluminum nitride precipitates are the most probable particles, which interact with the recovery and recrystallization processes during annealing. Their interactions and resulting effects on the microstructure and final properties strongly depend on the occurrence time of precipitation process with respect to different restoration processes. The sequence of precipitation and recrystallization processes in turn depend on the temperature and heating rate in the annealing process [11,26-28)]. At low temperatures in isothermal salt bath annealing, 550°C, the longer incubation time and lower recrystallization rate lead to simultaneous occurrence of precipitation and recrystallization and uncompleted recrystallization. At higher temperatures, e.g. 700°C, high recrystallization rate leads to complete recrystallization without significant effect of precipitates although precipitation process may occur during recrystallization. In MSP cycle with a very slow heating rate, Figure 10, there is enough time for occurrence of precipitation before recrystallization starts in cold worked material. Under slow heating conditions of box annealing process most precipitates are formed on original grain boundaries before recrystallization onset. Therefore, the original grain boundaries play an obstructing role on recrystallizing new grains leading to an elongated recrystallized grain structure [26,27)], Figure 5. Observation of non-homogeneous recrystallization, Figure 2d, suggests that interaction of recrystallization and precipitation processes is influenced by distribution of precipitates which, in turn, is dependent on the hot rolling conditions [29)]. The increase in hardness during annealing, Figure 8, is well consistent with the occurrence of precipitation. Lack of grain growth after recrystallization in samples annealed at 635 and 700°C, Figure 7, is attributable to the pinning effect of precipitates on grain boundaries at the final stages of recrystallization process or just after it. Long holding of samples at 700°C after recrystallization leads to the frequent observations of very large grains within small ones. These observations confirm the interaction of precipitation process and grain growth. Dissolution or coarsening of particles at high temperatures after long holding time leads to lose their pinning effect giving rise to abnormal grain growth or so-called secondary recrystallization as it is evident in Figure 4c.

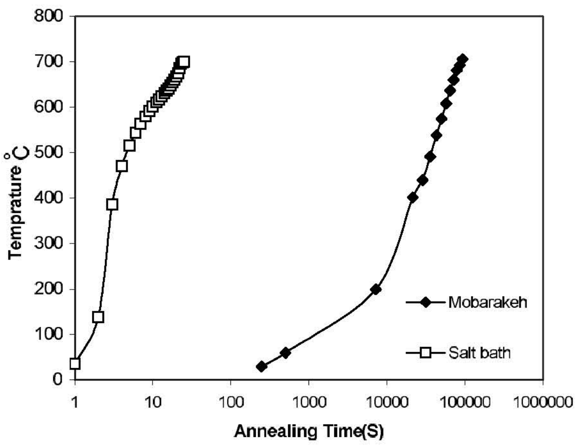

Fig. 10. Comparing the heating rate in isothermal salt bath annealing with MSP box annealing processes.

Conclusions

1- The heating rate during annealing has an important effect on the recrystallization behavior and final microstructure so that a slow heating rate produces elongated grains whereas a high heating rate leads to equiaxed grains.

2- Some precipitation processes occurring during annealing have profound effects on the recrystallization process. The sequence of recrystallization and precipitation processes, which is determined by heating rate, may lead to distinct different microstructures.

3- The interaction of precipitation process with recrystallization leads to relatively high apparent activation energy for recrystallization.

4- Hardness variations during annealing show a good consistency with the microstructural evolution and also indicate the effects of precipitation process.

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