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The Effect of Hot Deformation Parameters on Grain Size Refinement in a Martensitic Stainless Steel

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Abstract: The grain size refinement of AISI 422 martensitic stainless steel in the temperature range of 950-1150 ºC was investigated by hot deformation tests. The deformed specimens were held at deformation temperature with delay times of 5 to 300s after achieving a strain of 0.3. The austenite grains exhibit a considerable growth at temperature higher than 1050˚C, while the grain coarsening is negligible at lower deformation temperatures. Therefore, it is a difficult task to achieve a fine grain structure at these high deformation temperatures. In the second stage of this work, the grain growth behavior of the deformed alloy at temperature range of 940-1020 ºC was investigated to obtain a fine austenite grain in the final deformation step. A uniform and fine-grain structure, with average grain size less than 30 µm, can be obtained by considering the appropriate temperature and strain per pass. At 1020˚C, a relatively fine and uniform recrystallized grain, mean grain size of about 28 µm, is obtained with an applied strain of 0.4, while at 980˚C after strain of 0.2 a nearly equiaxed grain with the same mean grain size is achieved.

Keywords: Hot deformation, AISI 422 steel, Grain refinement, Recrystallization process.

1. Introduction

At relatively low operating temperatures (lower than 0.6 T_m K), the higher ductility and toughness can be attributed to the finer austenite grain size. This effect can be highlighted at long term exposure, where the small grain size is beneficial through hindering the formation of continuous and fragile films of carbide. These continuous films preferentially tend to form over the austenite grain boundaries, which can increase the probability of intergranular fracture [1].

The AISI 422 steel, as one of the high strength alloys, is widely used for gas turbine blades over long term service exposure [2, 3]. Therefore, a fine-grain microstructure is desirable to improve the mechanical properties of this steel. In this regard, a precise control of hot deformation parameters, namely, deformation temperature, strain per pass and number of passes is crucial for achieving a fine austenite grain structure. In addition, in martensitic steels, the coarser austenite grain will result in a coarser martensite structure, which has an adverse effect on plasticity, strength and toughness of the steels [4].

Generally, in hot forming processes (i.e. hot forging and hot rolling) the initial coarse-grained microstructure is replaced by fine recrystallized ones. However, depending on deformation parameters, the recrystallization phenomenon can result in grain refining or coarsening. The desirable grain refinement can be obtained through careful control of the recrystallization processes which is significantly dependent on deformation parameters [5].

In the area of grain refinement of forged alloys, Domblesky et al [6] have simulated multiple pass forging of Alloy 718 in the temperature range of 954-1066 ºC to investigate the effect of temperature, strain and interpass time (in the final deformation pass) on grain refinement of as-forged alloy. They found that

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the recrystallized grain size is independent of number of passes after the second deformation pass. Mataya et al [7] have investigated the microstructural evolution of Alloy 718 during multiple radial forging with considering the position of the billet (lead end, midlength and tail end) and their attribute interpass times. They concluded that the deformation temperature, strain per pass and interpass time can be considered as the major parameters affecting the final microstructural evolutions.

Several investigations have been carried out on the softening behavior and microstructural characterization of martensitic stainless steels [8- 11]. However, there are little published data regarding the effect of deformation parameters on the final grain size of these steels. Hence, in the current work, the recrystallized grain size of AISI 422 steel was investigated under single and double-hit deformation tests. For this purpose, the grain growth behavior was investigated over a temperature range of 950-1150 ºC for different interpass times (5 to 300 s). Subsequently, the effect of strain per pass (0.2, 0.3 and 0.4), interpass time (30, 60 and 120 s) and number of passes (single and double-hit deformation) were investigated on microstructural evolutions over the temperature range of 940 to1020 ºC.

2. Experimental procedure

The AISI 422 martensitic stainless steel with the following chemical composition: 0.21% C, 0.29% Si, 12.02% Cr, 0.62% Mn, 0.96% Ni, 0.96% Mo, 1.02% W (wt%) and Fe (balance),was used in the present study. According to ASTM A1033 standard, cylindrical specimens with 5 mm in diameter and 10 mm in height were prepared using an electrical discharge machining (EDM). The specimens were provided from the central part of the hot forged billet to ensure the uniformity of the structure.

Hot compression tests were carried out with BAEHR 805D/L dilatometer (BAEHR-Thermoanalyse GmbH) at strain of 0.2, 0.3 and 0.4 over temperature range of 950 to 1150 ºC.

The specimens were preheated up to 1200 ºC and held for 5 minutes for obtaining a homogenous austenitic structure. Then, specimens were cooled down to desirable deformation temperature and held for 20 to eliminate the thermal gradient prior to deformation. In the first part of the study, the recrystallization and grain growth behavior of the deformed alloy after a true strain of 0.3 was investigated over a temperature range of 950 to 1150 ºC after interpass times of 5 to 300. The second deformation step was applied with strain of 0.1 and finally the deformed alloy was immediately quenched to room temperature. The total cycle is schematically shown in Fig. 1. Subsequently, the effect of last deformation parameters on final austenite grain size was investigated. For this purpose, the hot compression tests were performed under strains of 0.2, 0.3 and 0.4 and temperature range of 940-1020 ºC. After the deformation step, the deformed specimens were unloaded and maintained for 30, 60 and 120 s. All the deformation tests were performed at a constant strain rate of 1 per second.

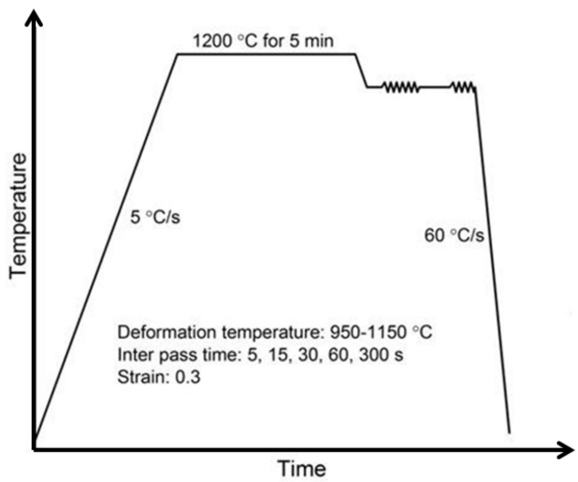

Fig. 1. Schematic illustration of compression tests.

The deformed specimens were cut through the longitudinal direction in order to study the microstructural evolution. The specimens were polished according to ASTME3 standard and the final polishing step was performed using colloidal silicon suspension. The electron-backscattered diffraction (EBSD) method was used to identify the austenite grain boundaries according to their misorientation. Then only those boundaries with misorientation in the range from 15 to 50 degrees with the step size of 350 nm were selected. In optical micrographs, the austenite grain boundaries revealed via a chemical etching solution with the following reagent: 3 g $CuCl_3$, 4 g $FeCl_3$, 10 ml HCl and 50 ml H_2O. The quantitative grain size measurement was carried out by Clemex Image analyzer.

3. Results and discussion

3.1 Effect of deformation temperature

In the first step of this work, the grain growth behavior in the temperature range of 950-1150 ºC was investigated after different strains and interpass times. It is apparent that the recrystallized grain continuously grows as holding time progresses. The final recrystallized-grain size increases with increasing the deformation temperature and interpass time. The growth of the initially recrystallized nuclei can be considered as the main process of microstructural variation during holding time, especially at higher temperatures.

A uniform and fine-grain structure (at least finer than the initial one) can be obtained by considering the appropriate temperature and holding time. For example, at 1150˚C a relatively fine and uniform recrystallized grain (mean grain size of about 32 µm) is obtained just after 5 s (Fig. 3a), while at 1000˚C after 300s a nearly equiaxed grain is achieved with an average grain size of 26µm (Fig. 3b). Fig. 4 illustrates the recrystallization progress of deformed specimens at 950 ºC with increasing holding times of 5, 30 and 300 s. From Fig. 4, it is clear that the new recrystallized grains nucleated during holding time and static recrystallization can be considered as their dominant softening mechanism. However, at deformation temperature of 1150 ºC (Fig. 3a), the recrystallization process is almost completed just after 5 s, which implies that the metadynamic recrystallization is the main restoration mechanism during holding time at this deformation temperature.

The final grain size is related not only to the deformation conditions but also to the holding times. It can be also concluded from Fig. 2 that the relatively coarse (more than 30 µm) austenite grain was obtained at deformation temperatures above 1050 ºC. At these high temperatures, it is a difficult task to achieve a fine-grain structure after the hot deformation process because the additional grain growth occurrs at the sufficiently high deformation temperature and enough holding time. In this regard, the grain growth behavior of the deformed alloy at deformation temperatures of 1020, 980 and 940 ºC (lower limit of hot deformation temperature) was studied to achieve a fine austenite grain size.

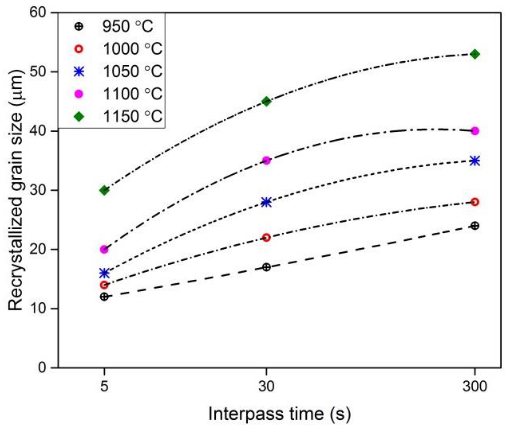

Fig. 2. Recrystallized grain size under different holding time at deformation temperatures of 1150, 1100, 1050, 1000 and 950 ˚C.

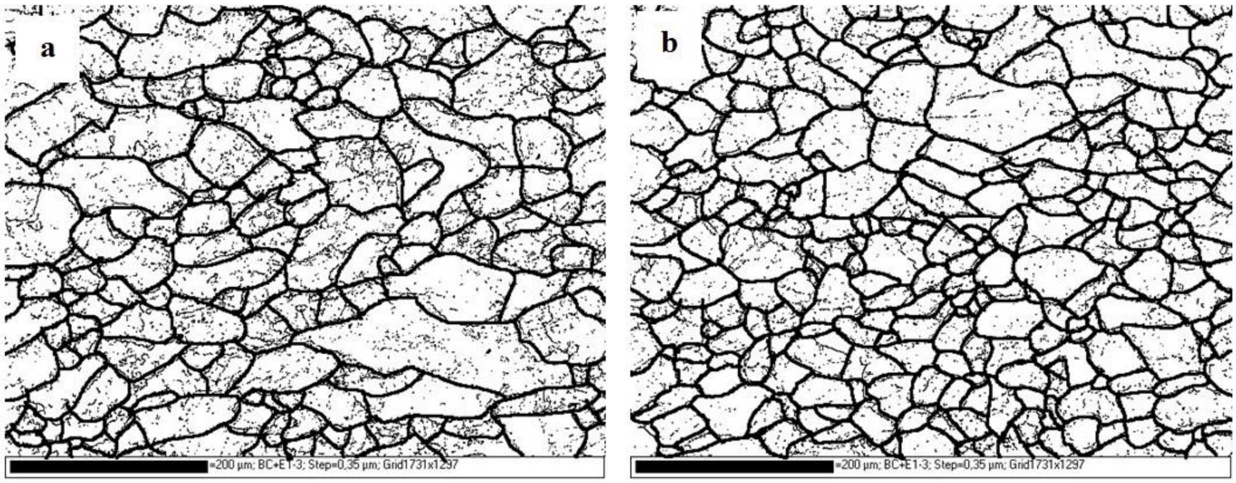

Fig. 3. EBSD observations show the austenite grain boundaries with misorientation in the range of 15º-50º at deformation temperature of a) 1150ºC after 5 s and b) 1000ºC after 300s.

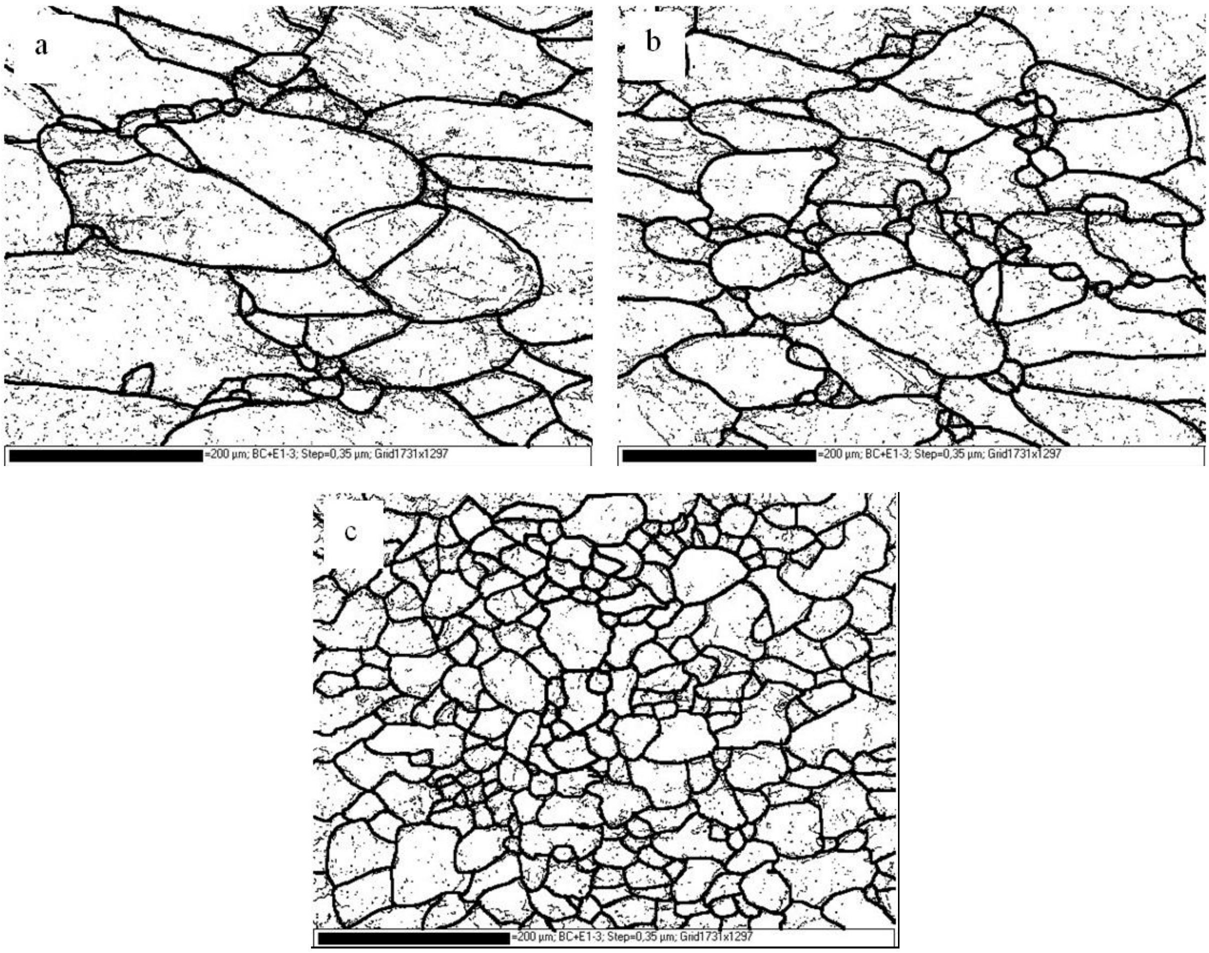

Fig. 4. EBSD observations illustrate the static recrystallization process of the deformed specimen at 950 ºC after 5 s b) 30 and c) 300s.

The flow stress curves of the studied steel at deformation temperatures of 940, 980 and 1020ºC up to true strain of 0.7 is shown in Fig. 5. Form the curves; it is clear that (up to strain of 0.4) no significant dynamic softening (recrystallization) is occurred. At relatively low deformation temperatures, lack of sufficient thermal energy for restoration processes and high strain rate are the reasons why dynamic softening is not remarkable.

The critical and peak point for initiation of dynamic recrystallization (DRX) was determined from the flow stress curves using Poliak and Jonas method [14, 15], which is based on changes in the strain hardening rate versus flow stress as drawn in Fig. 6. The $\varepsilon_C/\varepsilon_P$ (critical strain/peak strain) and σ_C/σ_P (critical stress/peak stress) were determined as 0.32 and 0.80 respectively, as given in Table 1. Also, these values for X20Cr13 martensitic stainless steel, with very close composition to the AISI 422 steel, tested under identical condition were reported as ($\varepsilon_C/\varepsilon_P$) 0.35 and ($\sigma_C/\sigma_P$) 0.92 [16]. Given the Table 1, in this study, the applied strains of 0.2, 0.3 and 0.4 are always greater than the critical strain for dynamic and static recrystallization. Therefore, we can expect the grain refinement of the deformed steel as the occurrence of metadynamic or static recrystallization process.

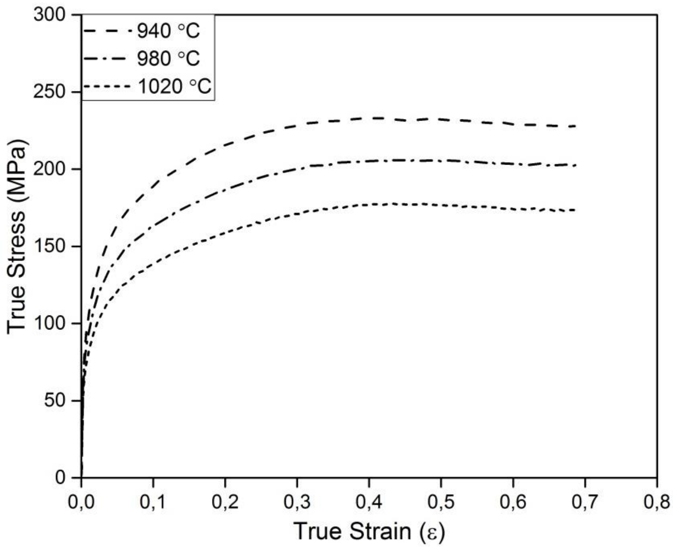

Fig. 5. Flow stress curves at deformation temperatures of 940, 980 and 1020ºC up to strain of 0.7.

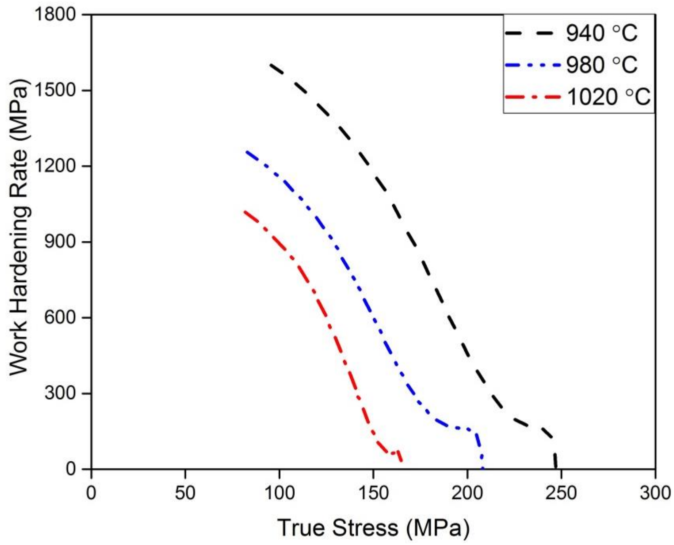

Fig. 6. Work hardening rate versus stress from yield to peak stress point.

Table 1. Stress and strain values at critical and peak point according to Fig. 6.

Temperature (˚C)	Critical Stress (σ_C)	Critical Strain (ε_C)	Peak Stress (σ_P)	Peak Strain (ε_P)	σ_C/σ_P	$\varepsilon_C/\varepsilon_P$
940	205	0.12	247	0.36	0.82	0.34
980	166	0.11	208	0.34	0.80	0.32
1020	129	0.09	165	0.30	0.78	0.30

3.2. Effect of strain per pass

The flow stress curves at deformation temperature of 940 ºC after strain of 0.2 and interpass times of 30, 60 and 120 s are illustrated in Fig. 7. It is clear that the flow stress is decreased as the interpass time increases, which is probably due to progress of static or metadynamic softening phenomena like recovery and recrystallization processes. The fractional softening is basically affected by deformation temperature, interpass time, and strain per pass [17-19].

The softening fraction which occurred during interpass time can be calculated by several methods [20]. In this work, the 0.2% offset stress method was used to estimate the softening fractions as follows:

$$\mathbf{X} = \frac{\boldsymbol{\sigma}_{\mathbf{m}} - \boldsymbol{\sigma}_{\mathbf{2}}}{\boldsymbol{\sigma}_{\mathbf{m}} - \boldsymbol{\sigma}_{\mathbf{1}}} \tag{1}$$

where σ_m is the final stress level prior to unloading, and σ_1 and σ_2 are 0.2% offset stress in the first and second deformation step, respectively. The calculated fractional softening for various deformation parameters are shown in Fig. 8. It is observed that the fractional softening increases at increasing deformation temperature and interpass time. However, the softening process is not completed at deformation temperatures of 940 and 980 ºC even after 120 s, whereas it nearly completed at temperature of 1020 ºC. Despite of the incomplete softening at deformation temperature of 980 ºC after 120 s, the microstructural observations show almost uniform distribution of austenite grain, as shown in Fig. 9. This contradiction can be attributed to progress of recrystallization process and reducs the size of unrecrystallized grains. Theses prior austenite grain size is in the range of recrystallized ones and it is difficult to distinguish between those. Therefore, the uniform distribution of austenite grain can be observed before completing the recrystallization process.

The fractional softening as a function of strain (0.2, 0.3 and 0.4) and deformation temperatures after 120s interpass time are plotted in Fig. 10. As it is clear, the fractional softening increases with increasing the strain and deformation temperature. McQueen and Jonas [21] concluded that the fractional softening of full recrystallized microstructure ranges from 90 to 120%, which attributed to grain refinement (90%) and coarsening (120%) comparing to the original grains. Therefore, according to Fig. 5, the softening process is almost completed at strain of 0.4 in all deformation temperatures. However, at lower applied strain, apparently the softening is not completed, despite the fact that the structure with fractional softening more than 80% has relatively uniform austenite grain. For example, the optical micrographs of the deformed specimen at 980 ºC and 940 ºC after 0.3 straining is shown in Fig. 11. A relatively uniform structure is obtained at deformation temperature of 980 ºC, whereas at 940 ºC the duplex structure (fine-recrystallized and coarse - unrecrystallized grains) is as the main microstructural characteristics of the specimen.

Figure 12 shows the recrystallized grain size after 120 s interpass time at strain range of 0.2 to 0.4 under deformation temperatures of 940, 980 and 1020 ºC. It is observed that the recrystallized grain size increases at increasing deformation temperatures and decreasing applied strain. For instance, at deformation temperature of 1020 ºC, the grain size decreases from 35 to 28 µm when increasing the strain from 0.2 to 0.4. In addition, under the same strain, the austenite grain size increases with increasing the deformation temperature. In this regard, the average recrystallized grain size has almost been doubled with increasing the deformation temperature from 940 ºC to 1020 ºC.

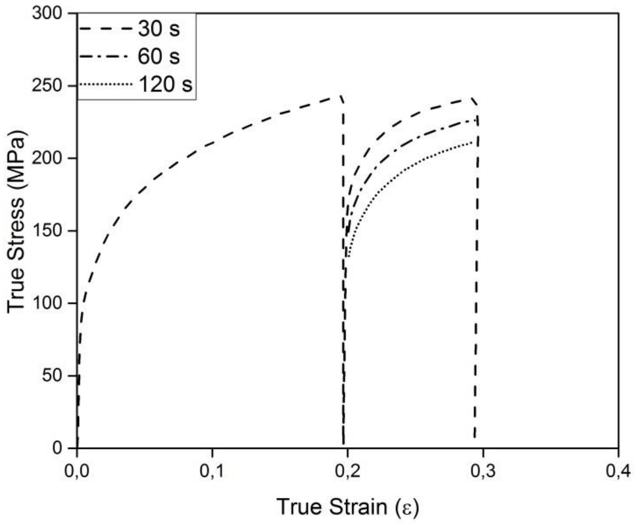

Fig. 7. Flow stress curves of deformed specimen at 940 ºC after inter pass times of 30, 60 and 120 s.

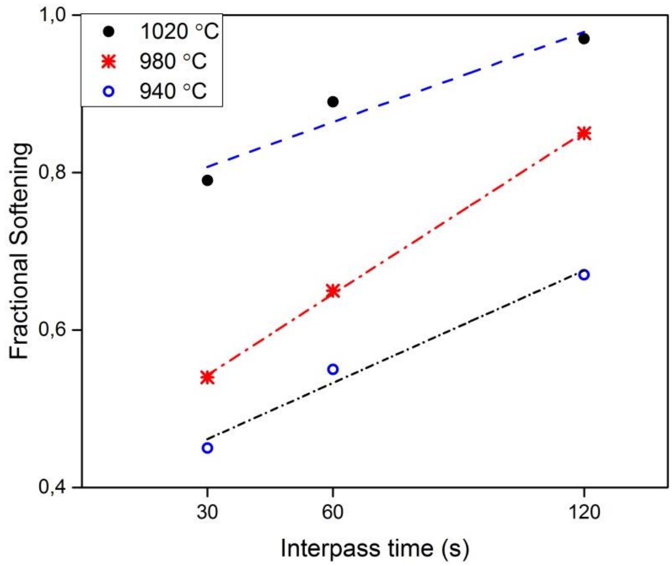

Fig. 8. Fractional softening at strain of 0.2 under interpass times of 30, 60 and 120 s.

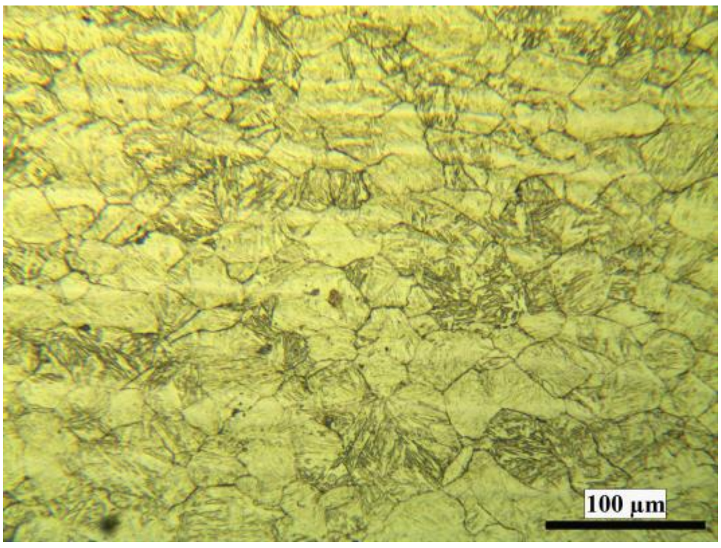

Fig. 9. Optical micrograph of deformed specimen at 980ºC and strain of 0.3 and holding for 120 s.

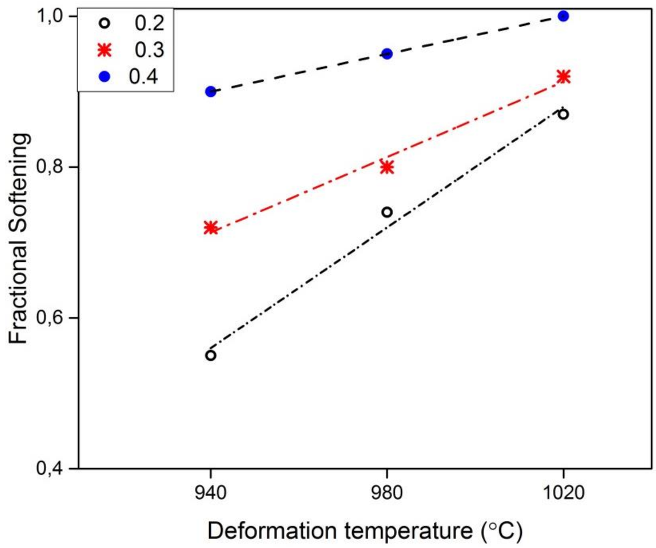

Fig. 10. Fractional softening after 120 s under true strain of 0.2, 0.3 and 0.4 at deformation temperatures of 940, 980 and 1020ºC.

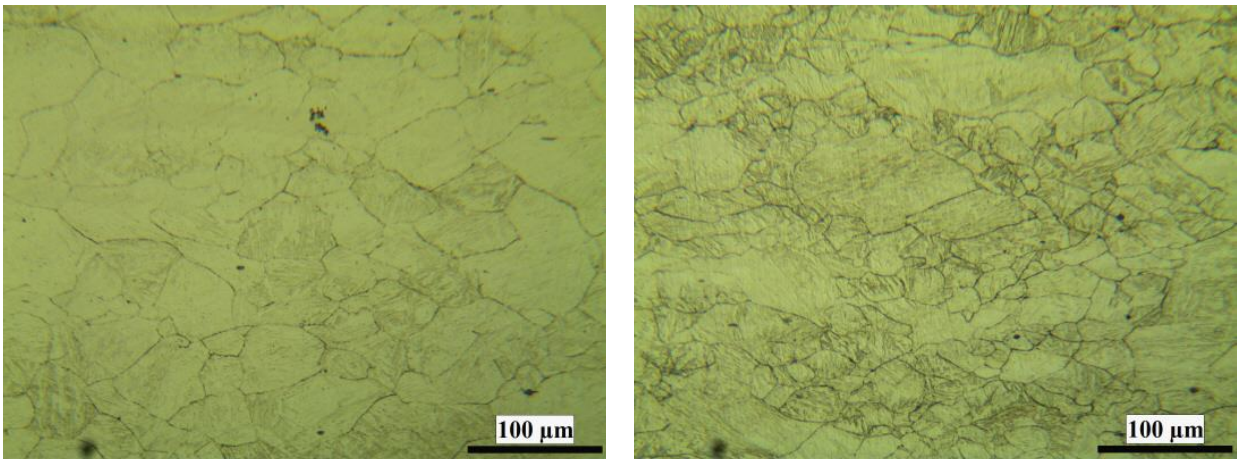

Fig. 11. Optical micrographs of deformed specimen at a) 980 ºC and b) 940 ºC after 120s.

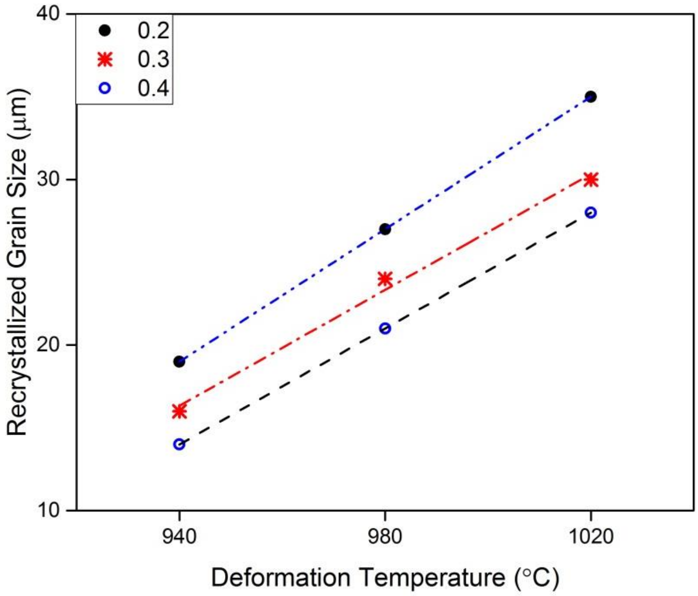

Fig. 12. Recrystallized grain size after 120 s interpass time at strains of 0.2, 0.3 and 0.4 under deformation temperatures of 940, 980 and 1020 ºC.

3.3. Effect of number of deformation pass

The double-hit hot deformation test at strain of 0.2 and 0.3 was performed to investigate the effect of number of deformation passes on microstructural evolution. The recrystallized grain size obtained in the single and double-hit deformation tests after 120 s of holding time are compared in Fig. 13. The recrystallized-grain size in double-hit deformation tests is always smaller than that of the single-hit deformation. As mentioned before, the grain refinement comparing to initial austenite grain size occurs at all deformation conditions. Therefore, the smaller prior austenite grain size is available in the second deformation pass. These excess grain boundaries can serve as nucleation sites for recrystallization of new grains, that results in smaller recrystallized-grain size compared to the single-hit deformation step.

In the final stage of this work, the recrystallized grain growth was investigated at longer holding time (i.e. more than 120 s). The recrystallized grain size at double-hit deformation test after 300 s (i.e. the average grain size) is illustrated in Fig. 14. Slow growth rate of recrystallized grain in the holding 120 to 300 s. is clearly shown. Therefore, it can be concluded that the final fine- austenite grain will result even at slow cooling rate at final stage.

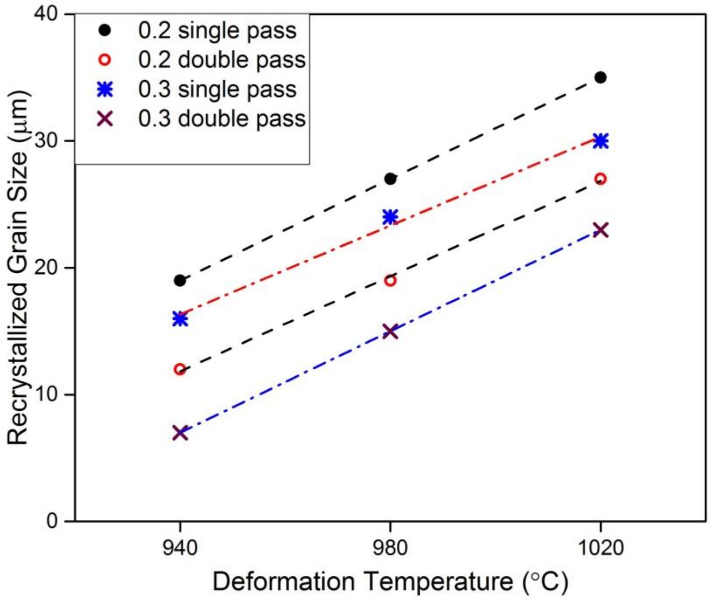

Fig. 13. Recrystallized grain size in single and double-hit deformation test under strain of 0.2 and 0.3 after 120 s.

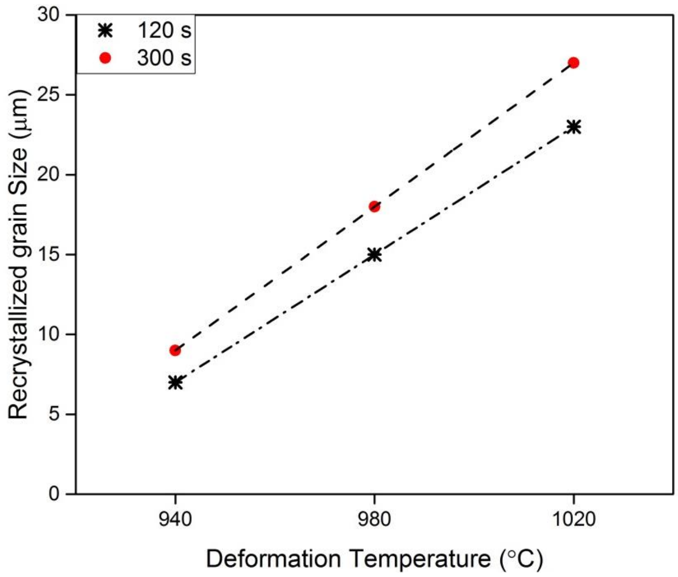

Fig. 14. Recrystallized grain size in double-hit deformation test under the strain of 0.3 after 120 and 300 s.

4. Conclusion

The effect of hot deformation parameters (strain, temperature and interpass time) on grain growth behavior of AISI 422 steel was investigated under a temperature range of 950-1150 ºC. In addition, the deformation parameters of final deformation pass were studied for achieving a fine austenite grain. The main points of this study are as follows.

1. The final recrystallized grain size is relatively coarse (more than 30µm) at deformation temperature higher than 1050 ºC. Therefore, the final deformation step must be conducted at lower temperatures to hinder the considerable grain growth in a relatively long time after deformation.

2. Static recrystallization is dominant grain refinement mechanism at deformation temperature of 950 ºC, whereas, the recrystallization process is progressed mainly metadynamically at 1150 ºC through holding time.

3. A uniform and fine-grain structure (at least finer than the initial one) can be obtained by considering the appropriate temperature, strain and holding time. For example, at higher deformation temperature, higher strain and shorter holding time is necessary to obtain the finer austenite grain.

4. The recrystallized-grain size in double-hit deformation tests is always smaller than the single-hit deformation ones.

5. At temperature range from 940-1020 ºC, a negligible grain growth is observed after double-hit deformation test in the holding times of 120 to 300 s. Therefore, it can be concluded that the fine-austenite grain would be obtained even at slow cooling rate to room temperature.

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بررسی اثر پارامترهای تغییرشکل داغ روی اصلاح اندازه دانه در فولاد زنگ نزن مارتنزیتی

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چکیده: در این مقاله با استفاده از آزمون‌های تغییرشکل داغ، رفتار رشد دانه‌های فولاد زنگ نزن مارتنزیتی AISI422 در محدوده دمایی 950-1150ºC مورد بررسی قرار گرفته است. نمونه‌های تغییرشکل یافته بعد از اعمال کرنش 0/3، در محدوده زمانی 300-5 ثانیه در دمای تغییرشکل نگهداری شده و سپس تا دمای محیط سرد شدند. بررسی رفتار رشد دانه‌های آستنیت نشان داد که در دماهای بالاتر از 1050ºC رشد دانه‌ها بسیار سریع بوده است در حالیکه در دماهای پایین‌تر، رشد دانه‌ها با سرعت محدودی انجام شده است. بنابراین دست‌یابی به یک ریزساختار ریزدانه در دماهای بالا، امری بسیار دشوار می‌باشد. از اینرو در قسمت بعدی این مقاله، به منظور حصول یک ریزساختار ریزدانه، رفتار رشد دانه‌های تبلورمجدد یافته در محدوده دمایی 940-1020ºC (به عنوان پاس نهایی تغییرشکل) مورد بررسی قرار گرفت. با درنظرگرفتن دمای تغییر شکل و مقدار کرنش اعمالی پاس آخر می‌توان به ریزساختاری ریزدانه و یکنواخت با اندازه دانه‌های کمتر از 30µm دست یافت. به عنوان مثال در دمای تغییر شکل 1020ºC و کرنش اعمالی 0/4، ریزساختاری یکنواخت با متوسط اندازه دانه‌های 28µm حاصل شده است در حالیکه ریزساختاری با همین اندازه دانه را می‌توان بعد از اعمال تغییر شکل در دمای 980ºC و کرنش اعمالی 0/2 بدست آورد.

واژه های کلیدی: تغییرشکل داغ، فولاد AISI422، ریزشدن اندازه دانه‌های آستنیت، فرآیند تبلورمجدد.