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Article *in* Materials Science Forum · July 2007

DOI: 10.4028/www.scientific.net/MSF.550.601

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Materials Science Forum Vol. 550 (2007) pp. 601-606
online at http://www.scientific.net

Kinetics, Mechanism and Modeling of Microstructural Evolution During Dynamic Recrystallization in a 15Cr-15Ni-2.2Mo-Ti Modified Austenitic Stainless Steel

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Keywords: Austenitic stainless steel; Dynamic recrystallization; Kinetics; Mechanism; Modeling; Artificial neural network.

Abstract. Kinetics, mechanism and modeling of the microstructural evolution of a 15Cr-15Ni-2.2Mo-0.3Ti modified austenitic stainless steel (alloy D9) during dynamic recrystallization (DRX) have been investigated. The kinetics of DRX has been investigated employing a modified Johnson-Mehl-Avrami-Kolmogorov (JMAK) model. The microstructural study shows that nucleation of new grains during DRX takes place on the parent grain boundary by a bulging mechanism. No significant texture component has been found to develop in the recrystallized matrix. A substantial amount of twins have been observed in the recrystallized matrix. It is proposed that twins play an important role during the nucleation and subsequent expansion of DRX in alloy D9, which in turn moderates the texture in the recrystallized matrix. An artificial neural network model has also been developed to predict the fraction of DRX and grain size, as a function of processing conditions. A good correlation between experimental findings and predicted results has been obtained.

Introduction

Austenitic stainless steels, primarily AISI 316 and its modifications, have been chosen world-wide as prime candidate materials for fuel cladding and subassembly wrapper tubes in fast breeder reactors. For the 500 MW(e) fast breeder reactor project (PFBR) in India, a 15Cr-15Ni-2.2Mo-0.3Ti austenitic stainless steel has been developed indigenously [1] for in-core applications. This conforms to ASTM A771 UNS 38660 and is commonly referred to as alloy D9. The material has to be processed through various hot forming techniques before it is fabricated into final components. During this processing, material undergoes shape change as well as changes in microstructure that depends on the process history. Therefore, it is of paramount importance to understand the kinetics and mechanism of microstructural evolution which, in turn, would help to achieve the required microstructure in the end product.

Alloy D9 is a low to medium stacking fault energy material and is expected to undergo dynamic recrystallization (DRX) during hot working. However, the DRX behaviour of alloy D9 is less studied and needs a detailed physical understanding and experimental evidence. Therefore, in the present study, kinetics and micro-mechanism of the dynamic processes in this alloy have been investigated by performing hot forming operations at different temperatures, strains and strain rates. The paper also discusses development of an artificial neural network model to correlate microstructural evolution of alloy D9 with the processing parameters.

Experimental

The chemical composition (in wt %) of alloy D9 investigated in this study is as follows: C-0.052, Mn-1.509, Si-0.505, S-0.002, P-0.011, Cr-15.05, Ni-15.068, Mo-2.248, Ti-0.032, B-0.001, Co-0.01, N-0.006, and Fe-balance. 30 mm height and 20 mm diameter specimens were forged in the temperature range 1223 K-1423 K in steps of 50 K in a 250-kg pneumatic hammer and in the temperature range 1223 K-1373 K in steps of 50 K in a 250-ton triple-action hydraulic press. The mean strain rate of the forge hammer and hydraulic press is 100 s^{-1} and 0.22 s^{-1} respectively. True strains of 0.1, 0.2, 0.3, 0.4 and 0.5 (in a single step) were imparted at each temperature in order to study the effect of strain. As soon as the operation was completed, the deformed specimen was water quenched within 2-3 s in order to freeze the deformed microstructure.

The rolling operations were performed in a 2Hi/4Hi-instrumented laboratory rolling mill (Carl Wezel Model No. 420/350/275). Tests were carried out in the temperature range 1173 K-1473 K (in steps of 100 K) at a roll speed of 16 rpm, and a true strain of 0.3 was achieved in a single step. The calculated mean strain rate for rolling operation was found to be 2.6 s^{-1}.

Characterization

The hot worked samples were cut along the longitudinal direction and one half of the sample was taken to prepare metallographic specimens. The microstructures were examined optically in the maximum deformation zone of the samples and grain sizes were measured employing linear intercept method. The fraction of recrystallization (%DRX) was evaluated after different working conditions, calculated based on hardness by employing the following equation:

$$\%DRX = \frac{H_{CW(x)} - H_{HW(x)}}{H_{CW(x)} - H_{SA}} \tag{1}$$

where $H_{CW(X)}$ denotes the hardness of the cold worked specimen at a strain level of x percent, $H_{HW(X)}$ is the hardness of the hot worked sample at same strain in a particular working temperature at which we want to find out the fraction of recrystallization, and H_{SA} denotes the hardness of solution annealed sample. To obtain $H_{CW(X)}$, cold working operation of alloy D9 at various strain levels were carried out by high strain rate compression testing machine at room temperature.

Results and Discussion

Kinetics. The Johnson-Mehl-Avrami-Kolmogorov (JMAK) model has been widely used to describe the kinetics of recrystallization process [2]. According to this model, the recrystallization behaviour at a given temperature should follow the relationship presented by Eq. 2:

$$X_v = 1\text{-}\exp(-kt^n). \tag{2}$$

In this equation X_v is the recrystallization fraction at any annealing time t, k is constant and n is the Avrami exponent or JMAK exponent. Experimental recrystallization kinetic measurements are usually compared with the JMAK model by plotting $ln(-ln(1- X_v))$ against $ln(t)$. According to Eq. 2, this should yield a straight line of slope equal to the exponent n. However, this JMAK model is better suited for static recrystallization, where the annealing time t is available. In the present study, some modification of the JMAK model has been carried out in order to investigate the kinetics of DRX. The t term was replaced byε, where ε is true strain. The modified JMAK model can be expressed by Eq. 3 as follows:

$$X_v = 1\text{-}\exp(-k\varepsilon^n). \tag{3}$$

Here X_v is the fraction of DRX which has been calculated employing Eq. 1. According to this modified model, if X_v *vs.* ε is plotted, it should yield a sigmoidal curve. The plot of X_v *vs.* ε for forge hammer (Fig. 1) shows sigmoidal curve. At high temperatures, the curves are steeper compare to those at low temperatures. This is because at high temperatures, the available thermal activation energy is greater, which helps the process to be completed in shorter times.

According to the modified JMAK model, a $ln(-ln(1- X_v))$ *vs.* $ln(\varepsilon)$ is plot should yield a straight line whose slope would be equal to Avrami exponent n. Figure 2 shows such a plot for a forge hammer operation that is straight line in nature. A similar relationship has also been observed for hydraulic press operations. So it can be corroborated that modification carried out in JMAK model is well supported by the experimental results.

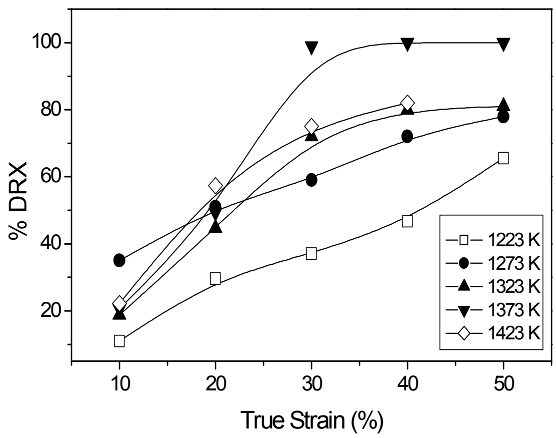

Fig.1: Effect of strain on DRX during forge hammer hammer

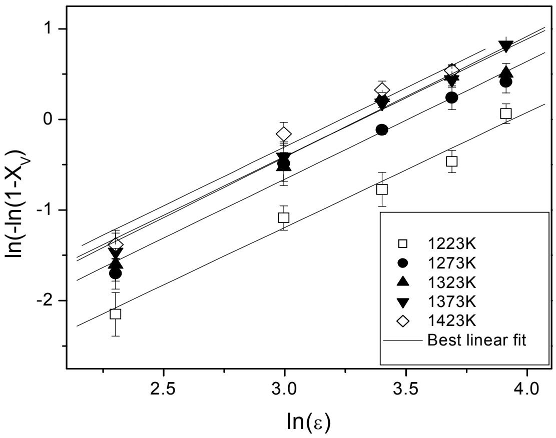

Fig.2: Modified JMAK plot for forge

The values of Avrami exponent (*n*) at different hot-working operations have been evaluated and it was found that the value of *n* varies in the narrow range 1.17-1.34. The variation of the Avrami exponent is associated with the transition from cyclic to single peak DRX [3]. A large value of *n* (~2) is an indication of cyclic DRX, whereas a low value of *n* (~1) conforms to single peak DRX. Therefore, based on the value of *n*, it could be suggested that alloy D9 exhibits single peak DRX. Sivaprasad *et al.* [4] have also reported single peak stress strain curve for this alloy during hot compression. The single peak DRX is also termed growth controlled DRX [5], where a large number of growing nuclei are formed and these growing nuclei mutually inhibit grain boundary migration. As a result grain growth is restricted, which eventually leads to grain refinement. In the present study, the grain refinement mechanism is clearly manifested in the microstructural investigation [Fig. 3].

Mechanism. At a temperature of 1223 K and low strain level, the microstructure consists of big grains (200 μm) and lamella like straight annealing twins [Fig. 3(a)]. The dynamically recrystallized grains are hardly found in the matrix. At the same temperature and a moderate strain level, the grain boundaries have become serrated in nature [Fig. 3(b)], which signifies the initiation of DRX in the deformed matrix. The curvy twin surfaces also indicate the initiation of DRX. The bulging of parent grain boundary and subsequent evolution of new DRX grains is clearly manifested in Fig. 3(c). This signifies that nucleation of DRX takes place in the parent grain boundary by bulging mechanism. With increasing temperature, many DRX grains appear in the deformed matrix and incomplete necklace structures developed [Fig. 3(d)]. At a temperature of 1373 K and 0.3 strain level, as shown by Fig. 3(e), more than half of the deformed matrix has been consumed by new DRX grains. Finally, at maximum temperature and strain level, the deformed grain structures have completely been disappeared and the microstructure consists of small equiaxed grains with an average grain size of 45 μm [Fig. 3(f)]. It can be noticed here that the fraction of DRX in the deformed matrix, manifested in the optical micrograph, is showing a similar kind of trend as found in Fig. 1, calculated based on hardness measurements employing Eq. 1. So, it could be suggested that the hardness measurement method can be successfully applied to calculate the recrystallized fraction.

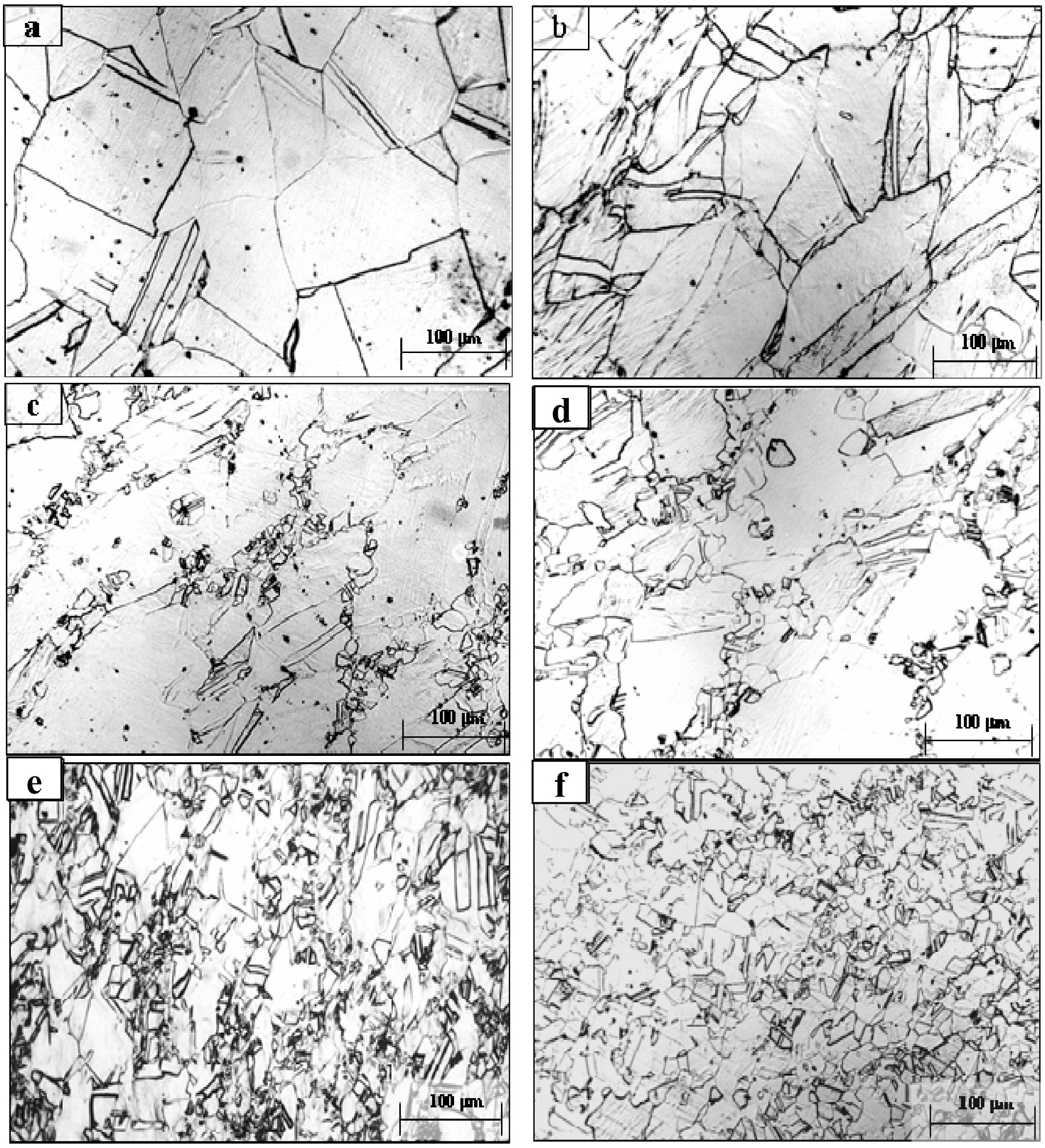

Fig.3: Optical micrograph of alloy D9 (a) forge hammer: T = 1223 K, ε = 0.07 (b) hydraulic press: T = 1223 K, ε = 0.21 (c) forge hammer: T = 1223 K, ε = 0.28 (d) forge hammer: T = 1273 K, ε = 0.28 (e) rolling: 1373 K, ε = 0.3 (f) forge hammer: T = 1423 K, ε = 0.46

The bulging mechanism is able to describe how the first recrystallized grains and, the first layer of new recrystallized grains around the prior grains form. However, this mechanism can not account for the expansion of the necklace structure throughout the deformed matrix. This is because, in the course of DRX when pre-existing grain boundaries are entirely covered by new grains (site saturation), bulging would have to proceed from the small recrystallized grains, which requires a very high boundary curvature. This makes further nucleation by bulging unlikely, because the very high driving force necessary to offset the high surface tension of the bulge is not available in a hot deformed microstructure [6].

The most crucial step for nucleation of DRX in the deformed matrix is the generation of a mobile grain boundary. Mobility increases with increasing misorientations, but a 10-15° misorientation is commonly assumed to be necessary. From the orientation image microscopy (OIM) maps and corresponding misorientation distribution plot [Fig. 4], it can be seen that misorientations in excess of 10° were occasionally formed. On the other hand an appreciable level of high misorientaion boundaries (>55°) are observed in the matrix, which could be accounted for twin boundaries [7]. The substantial level of twining is also observed in optical micrograph. From the electron back scattered diffraction studies (EBSD), it has been observed that micro-texture of the recrystallized volume is virtually random. This randomization of texture could be attributed to the repeated twining in the deformed matrix. Twining results in the formation of new grains with 60, 180 or 300° rotated orientation around <111> axis with the parent grains. As all the directions except the direction of the rotation axis are changed by the rotation operation, this means that no significant texture sharpening is expected unless the stable orientation of deformation is {111} [8]. However, the main component of texture developed in fcc metals is not {111}, but {011} [9]. Further, Gottstein has also reported that multiple twining results in a randomization of texture in the matrix [10]. So, from the present observations, it seems twining may play an important role during the nucleation and subsequent expansion of DRX in alloy D9, which in turn randomizes the texture in the recrystallized matrix.

Neural network results. A multilayer perceptron based feed-forward neural network has been employed to predict the microstructural evolution as a function of processing conditions. The inputs of the model are strain, strain rate and temperature, whereas microstructural features namely %DRX and average grain size is the output. The network has been trained and subsequently tested by the database obtained from the present study. Instead of standard back propagation (BP) algorithm, the network has been trained using some advance BP type algorithms such as resilient propagation (Rprop) and superSAB. It has been observed that an ANN with 12 hidden neurons in a single hidden layer produces best performance for %DRX prediction. However, the superSAB algorithm using 10 hidden neurons produces best performance for average grain size prediction. The comparison between experimental and predicted data for %DRX and average grain size prediction has been shown in Fig. 5(a) and Fig. 5(b) respectively. It could be observed that agreement is good throughout the whole range. Therefore, the model can be applied to predict the microstructural evolution during industrial scale metal forming processes of alloy D9 with sufficient accuracy and reliability.

Conclusions

Kinetics and mechanism of DRX has been investigated in a 15Cr-15Ni-2.2Mo-0.3Ti modified austenitic stainless steel (alloy D9). The kinetics of DRX have been studied using a modified JMAK model represented by the Eq. $Xv = 1\text{-}exp(-k\varepsilon^n)$. The experimental recrystallization kinetics measurements of alloy D9 have been found to agree with the modified JMAK model. The value of Avrami exponent found to lie in the narrow ranged 1.17-1.34, which signifies that alloy D9 exhibits growth controlled DRX. The microstructural study shows that nucleation of new grains during DRX takes place on the parent grain boundary by bulging mechanism. No significant texture component has been developed in the recrystallized matrix, as revealed by EBSD. This may be attributed to twin boundaries that constrain the orientation of the grains and thereby reduce the texture. A substantial amount of twining has also been identified in DRX matrix by optical micrograph and OIM. Therefore, it seems twins may play an important role during the nucleation and subsequent expansion of DRX in alloy D9, which in turn moderate the texture in the recrystallized matrix. An artificial neural network model has also been developed to predict the microstructural features, namely fraction of DRX and grain size, at different processing conditions. A good correlation between experimental findings and predicted results has been obtained. An instantaneous microstructure, therefore, can be designed in order to optimize the process parameters based on the microstructural evolution.

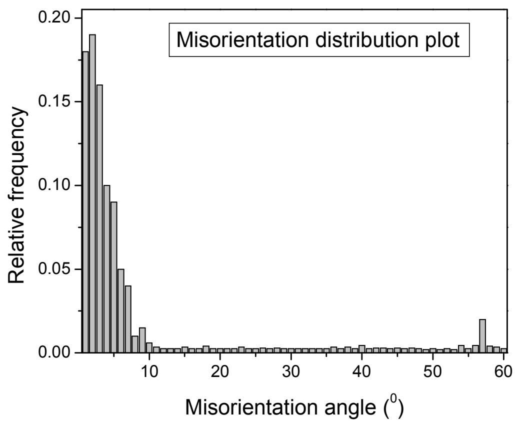

Fig.4: Misorientation distribution plot of forge hammered sample (T = 1273 K & ε = 0.2)

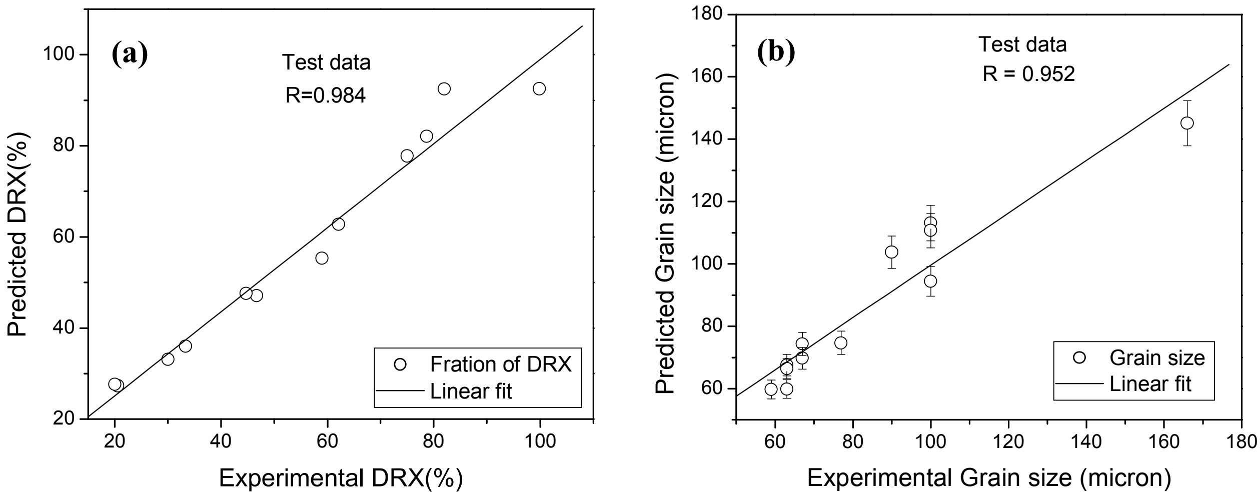

Fig.5: Comparison between experimental and ANN predicted for (a) % DRX and (b) grain size

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