

Characterization of Microstructural Changes During Annealing of Cold Worked Austenitic Stainless Steel Using Ultrasonic Velocity Measurements and Correlation with Mechanical Properties

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A technique that is sensitive to change in texture is more reliable than mechanical property measurements for the study of recrystallization behavior in alloys where recrystallization is coupled with precipitation. Hence, ultrasonic velocity measurements have been employed to characterize microstructural changes during annealing of cold worked Ti-modified austenitic stainless steel where precipitation of TiC is known to retard recrystallization. Mechanical property measurements, such as strength and hardness, could not distinguish the recovery and recrystallization regimes, and it was difficult to determine the exact time or temperature for onset of recrystallization. The variation of velocity with annealing time or annealing temperature exhibited a three-stage behavior. The velocity exhibited a slight increase in the recovery region followed by a sharp and continuous decrease in the recrystallization region and reached saturation on completion of recrystallization. Based on the microstructural investigations, the three stages were identified to be recovery, progress of recrystallization, and completion of recrystallization. Velocity measurements could sense the onset, progress, and completion of recrystallization more accurately compared with that of hardness and strength measurements.

Keywords annealing, austenitic stainless steel, cold work, mechanical properties, ultrasonic velocity

1. Introduction

The properties, state, and special characteristics of a given material should be considered while choosing a particular technique for studying annealing behavior. Recovery, recrystallization, and grain growth are the three annealing processes that bring out the changes in the cold worked microstructure. Among these processes, recrystallization is the microstructural process by which new strain-free grains form from the deformed microstructure. Depending on the material, recrystallization is often accompanied by other microstructural changes such as recovery, decomposition of solid solution, precipitation of second phases, phase transformations, and so forth. These changes influence the course of the recrystallization and often mask the picture revealed by certain methods of analysis. Direct methods such as metallography and x-ray structural analysis and indirect methods based on variations in the mechanical and physical properties are generally used for studying recrystallization behavior of materials. Structural methods such as x-rays can determine the onset of recrystallization more accurately compared to any other technique.^[1] The techniques based on x-rays have several variations, all of which are sen-

sitive to the detection of new strain-free grains, but accuracy and reproducibility are poor when quantitative information is required concerning the extent of recrystallization after any given annealing treatment. Moreover, x-ray analysis is restricted to a surface depth of maximum 40 μm only. Indirect methods for determining the temperature and time for beginning and end of recrystallization are based on sensing the changes in the structure-sensitive properties that react strongly to the variations in the density and distribution of dislocations that take place during recrystallization. These properties include hardness measurements, strength measurements, electrical conductivity, internal friction, and so forth. These methods become less reliable if recrystallization is coupled with phase transformations or precipitation. These indirect methods often give only approximate results. It is difficult to determine the actual onset and completion of recrystallization using the common methods in heterophase and precipitation hardening alloys.

The most common techniques used to study annealing behavior of metals and alloys are hardness testing and optical metallography. The techniques of hardness measurement and optical metallography are often found to give different values for the temperature or the time at which the recrystallization process either starts or is completed.^[2] Quantitative metallography techniques that are often used to measure the extent of the recrystallization process are time consuming and error prone. A new method based on thermoelectric power measurements was reported to be a valuable technique in studying recovery and recrystallization behavior in some metals and alloys.^[3] The use of this technique is, however, practically restricted to those materials that possess a large phonon drag near room temperature. Also, study of recovery and recrystal-

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Table 1 Chemical Composition (wt.%) of the Ti-Modified Austenitic Stainless Steel

C	Ni	Cr	Mo	Mn	Ti	Si	N	S	P	Fe
0.052	15.068	15.051	2.248	1.509	0.315	0.5	0.006	0.0025	0.011	Balance

lization for the alloys that exhibit simultaneous precipitation and textural changes is not possible using this technique. The techniques based on the physical and mechanical property variations cannot distinguish clearly the recovery, recrystallization, and grain growth regimes. The actual temperature or time for the onset and completion of recrystallization cannot be determined by using the above techniques. However, the actual onset and completion of recrystallization can be determined in materials if a technique is sensitive to the changes in texture brought by recrystallization. Though techniques based on x-rays are sensitive to changes in texture, their measurements are restricted to the surface only.

Ultrasonic velocity measurements have been successfully used to characterize microstructural changes during hardening and tempering in steels, age hardening in aluminum alloys, and to estimate grain size in austenitic stainless steels and other alloys.^[4-7] T. Jayakumar^[8] has used ultrasonic velocity measurements for estimating the degree of cold work in an austenitic stainless steel. Because recrystallized microstructure exhibits a different grain texture compared to unrecrystallized microstructure in materials and thus possesses different elastic constants, the measurement of ultrasonic velocity has the potential for determining the degree of recrystallization. Also, the technique enables volume measurements averaged throughout the thickness of the specimen. Edward R. Generazio^[9] successfully used ultrasonic attenuation measurements for determining the onset, degree, and completion of recrystallization in superalloys. Richard C. Stiffler et al.^[10] have employed the ultrasonic velocity measurement technique using horizontally polarized shear waves for determining the degree of recrystallization in an aluminum sheet. M. Vasudevan et al.^[11] first reported the use of ultrasonic thickness meter (velocity measurements) for characterizing annealing behavior in a cold worked austenitic stainless steel. Vasudevan et al.^[12-14] also reported the superiority of ultrasonic velocity measurements over mechanical property measurements to measure precisely the time and temperature for onset of recrystallization in this type of material.

It is important to study the recrystallization behavior of 15Cr-15Ni-2.2Mo-Ti-modified austenitic stainless steel, as this material in the 20% cold worked condition is the reference material for the clad and wrapper components of the prototype fast-breeder reactor in India.^[15] Exposure at elevated temperatures during service would degrade the cold worked microstructure due to recovery and recrystallization. Degradation in the cold worked microstructure in turn would decrease the resistance of the material against irradiation damage. In the cold worked condition, precipitation of TiC is reported to interact strongly with recovery and recrystallization processes, and, hence, use of common techniques to study recrystallization behavior can only give approximate results. In the current investigation, precise ultrasonic velocity measurements (based on the pulse-echo overlap principle) have been carried out in

the frequency range 2-20 MHz to characterize the annealing behavior of cold worked titanium-modified austenitic stainless steel. Ultrasonic velocity values were correlated with the microstructures obtained using optical metallography. Velocity measurements have also been compared with that of hardness and strength measurements.

2. Experimental Procedure

The chemical composition of the material used in the present investigation is given in Table 1. The material was initially in the form of an 11 mm diameter rod. The rod was solution-annealed at 1343 K for 0.5 h. The grain size after annealing was found to be in the range 20-30 μm . The rods were deformed in tension in an Instron 1195 universal testing machine to impart 20% cold work. Specimens cut from the cold worked rods were isothermally annealed at temperatures 923, 1023, 1073, and 1123 K for times in the range 0.5-1000 h. Vickers hardness was measured using a load of 10 kg. A minimum of five measurements were taken for each aging condition. Isochronal annealing was carried out in the temperature range 873-1173 K for times up to 1, 100, and 1000 h. Room-temperature tensile and hardness testing were carried out for the isochronally aged samples. Yield strength values were determined from the load elongation plots and the dimensions of the specimen. Microstructural changes were studied using optical metallography. Onset and completion of recrystallization was confirmed by estimating the volume fraction recrystallized using quantitative metallography. X-ray diffraction (XRD) data was obtained to provide evidence for the change in texture during recrystallization.

Ultrasonic velocity measurements were carried out on the 20% cold worked and annealed specimens by using both longitudinal wave probes (2, 10, and 20 MHz). The probes are of normal beam and broad band type. Filtered machine oil was used as the couplant for the longitudinal wave probes. A number of methods are available for precise transit time measurements. In the current work, a pulse-echo overlap^[16] instrument developed at our center was used. This method gives most accurate measurement of ultrasonic wave velocity. The accuracy arises from the fact that the method is capable of precisely measuring the transit time from any cycle of one echo to the corresponding cycle of the next echo. The principle of measurement involves making two successive or selected back-echo signals of interest to overlap on an oscilloscope by driving the x -axis with a carrier frequency whose period is the travel time between the signals of interest. Then, one signal appears on the first sweep of an oscilloscope and the other signal appears on the next sweep. Once the overlap of the selected back-echo signals is achieved, accurate transit time is obtained from the digital display. A complete description of the method with sketches is reported elsewhere.^[7] Following the above procedure, measurements of transit time were made on all the

specimens. By measuring the specimen thickness precisely (± 1 m), μ precise ultrasonic velocity values (± 3 m/s) were obtained. Totally, five measurements were made on each specimen, and the average value of the ultrasonic velocity was obtained for each specimen. More measurements enhanced the wave-microstructure interaction process, and the measured ultrasonic velocity values were representative of the bulk of the material.

3. Results and Discussion

The velocity, hardness, and strength values measured for the cold worked condition are marked on the abscissa of the figures respectively showing the variation of velocity, hardness, and

Table 2 Ultrasonic Velocities (m/s) in Different Material Conditions at 2, 10, and 20 MHz Longitudinal Wave Frequencies

Material Condition	Ultrasonic Velocities (m/s)		
	2 MHz	10 MHz	20 MHz
20% cold worked	5883	5879	5877
Recrystallized	5771	5738	5731

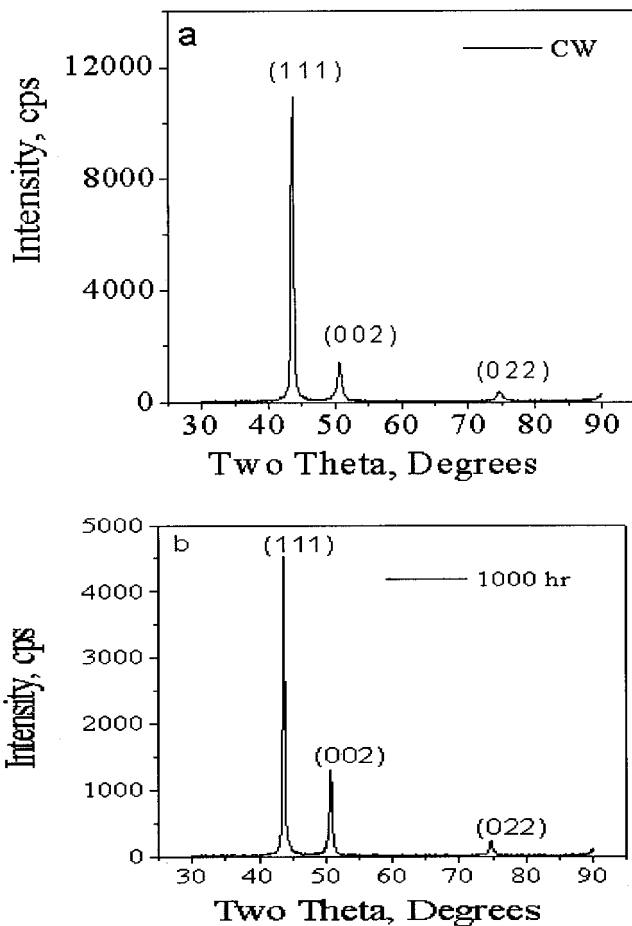
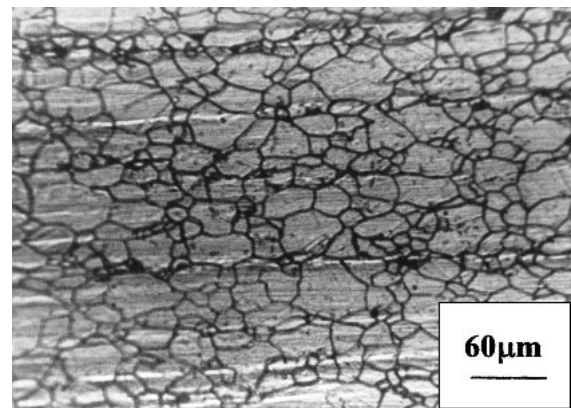
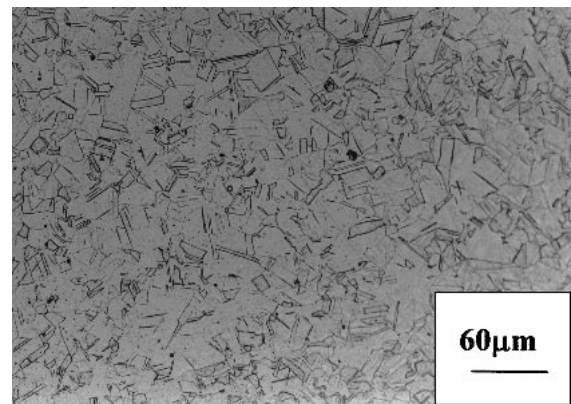


Fig. 1 X-ray diffraction data for the (a) 20% cold worked and (b) recrystallized conditions



(a)



(b)

Fig. 2 Optical microstructures of the (a) 20% cold worked and (b) recrystallized specimens

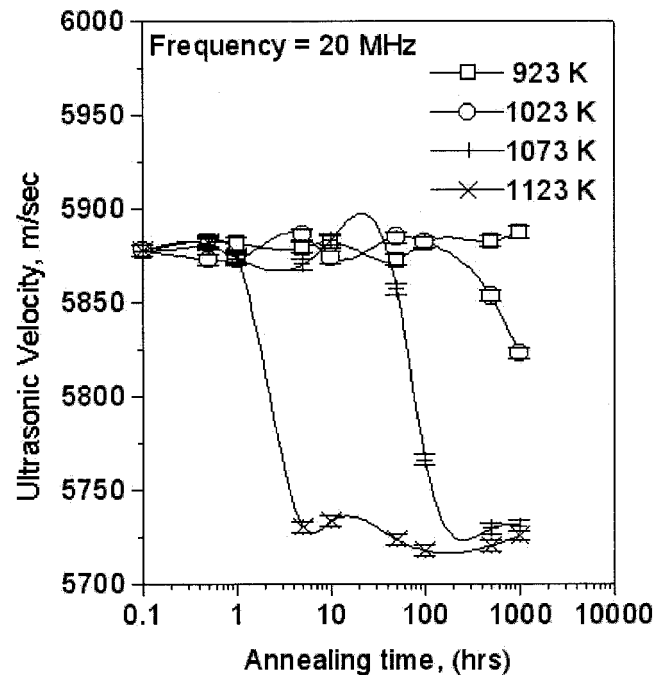


Fig. 3 The variation of ultrasonic velocity with annealing time at different annealing temperatures

strength with annealing time or temperature. The ultrasonic velocity values measured on the 20% cold worked and recrystallized samples using longitudinal waves at 2, 10, and 20 MHz frequencies are given in Table 2.

There is a difference of 2.5% (about 100-140 m/s) between the velocity values measured on the samples in the different material conditions (i.e., cold worked and recrystallized conditions). In order to find out the reason for the large difference in the velocity values, XRD patterns for cold worked and completely recrystallized samples were obtained. Figures 1(a) and (b) show the XRD patterns for the cold worked and recrystal-

lized samples and Fig. 2(a) and (b) show the optical microstructures of the 20% cold worked and recrystallized samples, respectively. There is a definite change in the intensity of the (111) reflection during annealing at 1073 K. The intensity of this reflection decreased by more than 50% due to recrystallization. The texture change observed by x-ray diffraction caused the drastic decrease in the ultrasonic velocity during recrystallization. Hence, the sensitivity of ultrasonic velocity to the occurrence of recrystallization in the 20% cold-worked stainless steel is attributed to the decrease in the intensity of (111) reflections as observed in the XRD pattern.

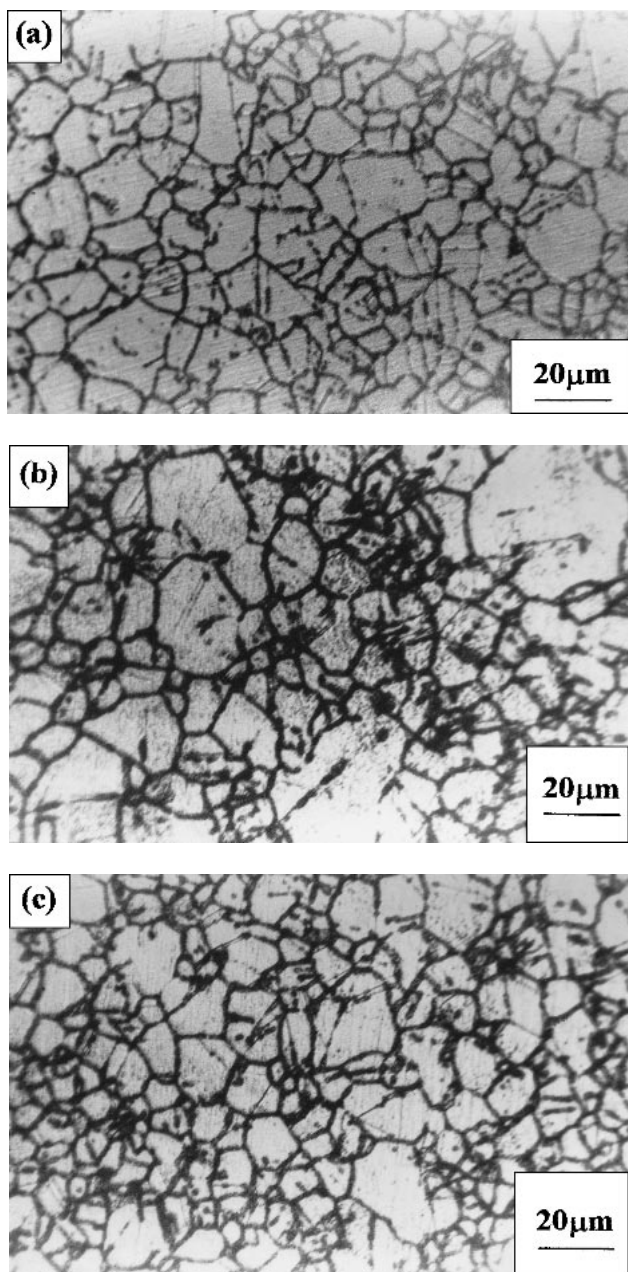


Fig. 4 Optical microstructures of the specimens annealed at 923 K for time (a) 50 h, (b) 500 h, and (c) 1000 h

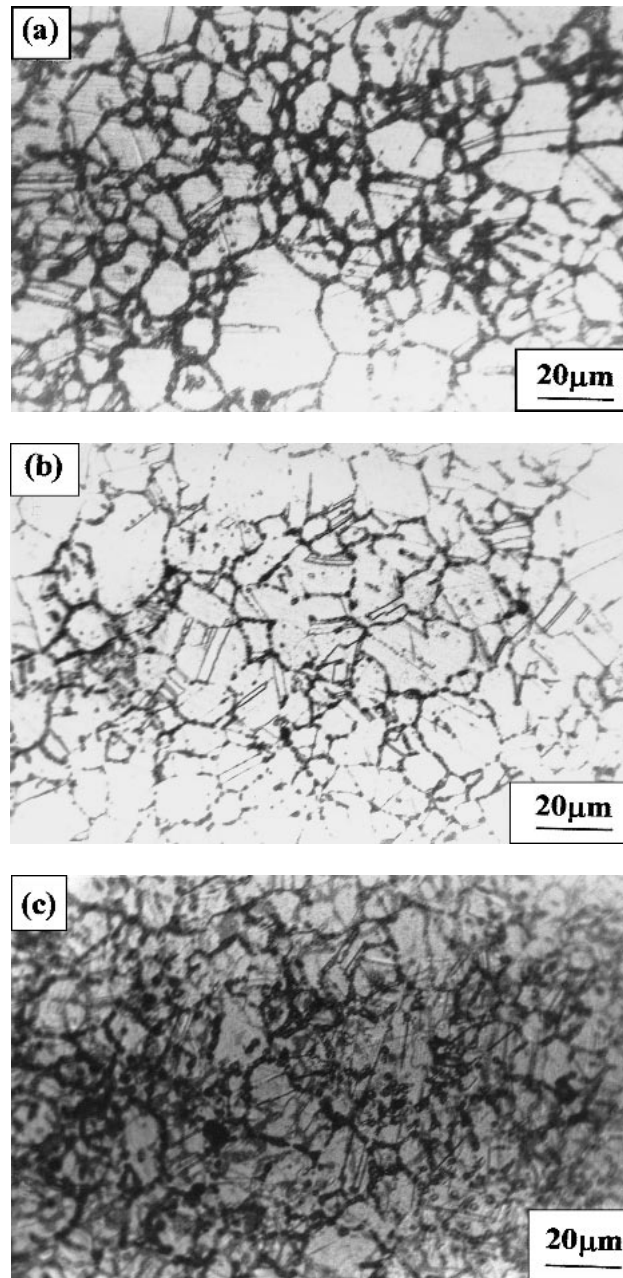


Fig. 5 Optical microstructures of the specimens annealed at 1023 K for times (a) 50 h, (b) 500 h, and (c) 1000 h

3.1 Isothermal Annealing Behavior

The variation of ultrasonic velocity (20 MHz) with annealing time at different temperatures is shown in Fig. 3. Variation of velocity with annealing time for the other frequencies (2 and 10 MHz) are reported below in the section on frequency effect. The precipitation of TiC (size in the range 10-20 nm) at a temperature of 923 K and above in the cold worked 15Cr-15Ni-Ti-modified austenitic stainless steel has been reported widely in the literature.^[17-19] Microstructures of the specimens annealed at 923 K is shown in Fig. 4. Precipitates are not seen in the optical microstructures because of their smaller size (15-30 nm). Hence, the combined effect of recovery and precipitation of TiC is only reflected in the variation of velocity with annealing time at 923 K. At 923 K, the variation of velocity with annealing time remains almost constant (Fig. 3). However, a definite increase in the velocity (about 30 m/s) value with annealing time for the specimens annealed at 923 K is seen at the frequency of 2 MHz. Also in our earlier paper,^[11]

we have reported an increase in velocity (2 MHz) in the recovery region, and the increase in the velocity was attributed to a decrease in the distortion of the lattice caused mainly by the annihilation of point defects and dislocations of opposite signs. In aluminum alloys, a definite increase in the ultrasonic velocity due to a decrease in the distortion of the lattice during aging has been reported.^[4,5] The increase in the velocity due to recovery is not seen at the frequency of 20 MHz in the current study because the ultrasonic waves at high frequencies (>10 MHz) have relatively shorter wavelengths, and, hence, the ultrasonic waves become sensitive to fine precipitates and are expected to interact with them. This interaction between the precipitates and the ultrasonic waves with shorter wavelengths (at high frequencies) could suppress the possible increase in the velocity due to recovery. There is no decrease in velocity even after 1000 h of annealing at 923 K, which implies that there is no change in the texture and hence no recrystallization. There is also no sign of recrystallization in the microstructures of the specimens annealed at 923 K (shown in Fig. 4). The decrease

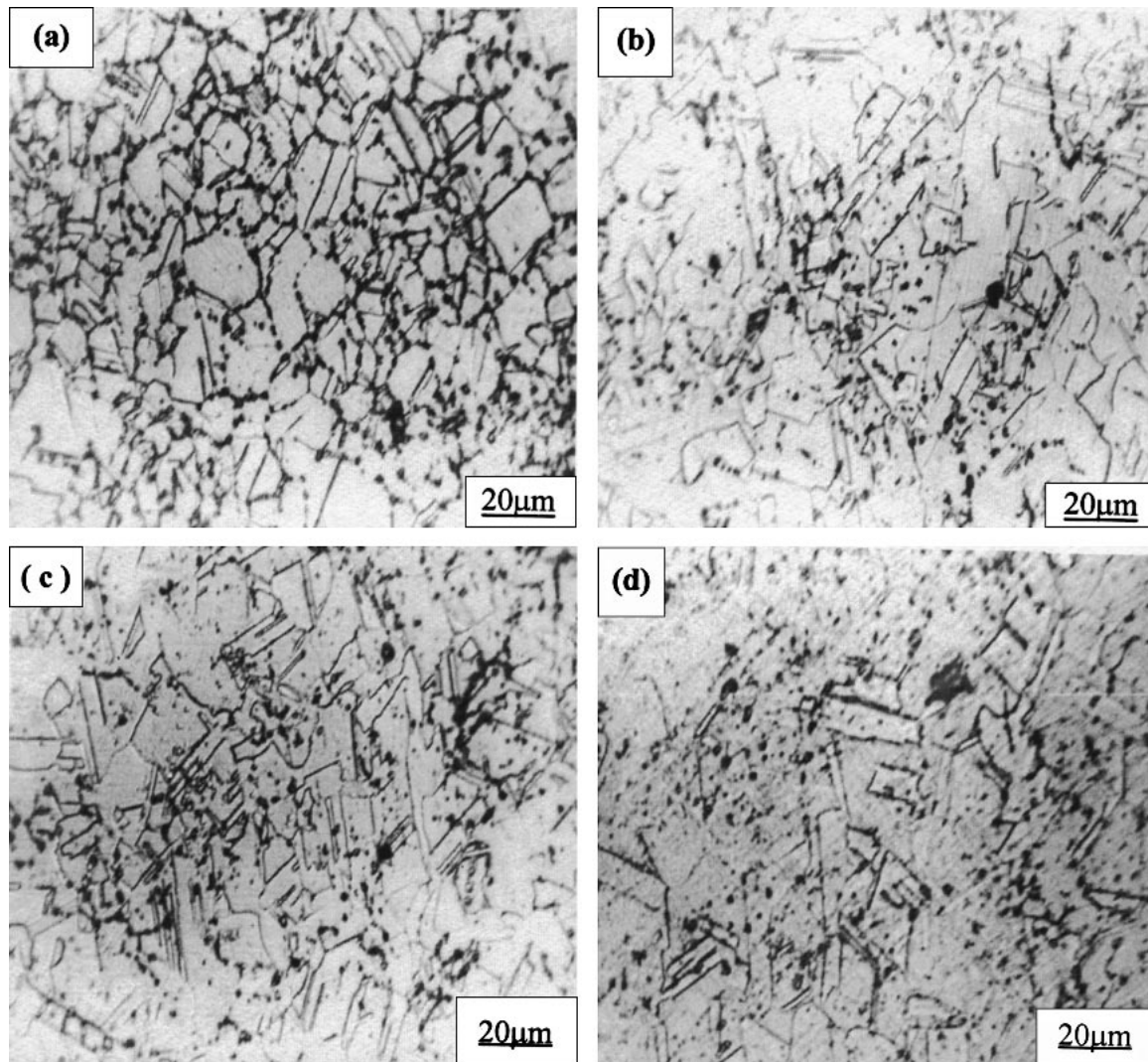


Fig. 6 Optical microstructures of the specimens annealed at 1073 K for times (a) 10 h, (b) 50 h, (c) 100 h, and (d) 500 h

in the velocity observed above 100 h of annealing at 1023 K is attributed to the change in the texture caused by recrystallization. The variation of velocity with annealing time exhibits no saturation even after 1000 h of annealing, which implies that the recrystallization is not yet complete. Figure 5 shows the microstructures of the annealed specimens at 1023 K. The microstructure (Fig. 5a) after 50 h of annealing shows no recrystallization while the microstructures (Fig. 5b and c) after 500 and 1000 h of annealing show retardation of recrystallization by extensive precipitation at the grain boundaries and the interior of the grains. Precipitation of $M_{23}C_6$ and TiC at the grain boundaries and precipitation of TiC at the grain interiors have been reported in the literature.^[19] The retardation of recrystallization by extensive TiC precipitation at 1023 K is consistent with the fact that the nose temperature in the time-temperature-precipitation diagram for this type of material is also 1023 K.^[20,21] Hence, recrystallization is only partial even after 1000 h of annealing. There is excellent correlation between the microstructural changes during annealing and the velocity measurements.

At 1073 K, the variation of velocity with time exhibit a three-stage behavior. In the first stage, a slight increase in the velocity value is noticed beyond 5 h of annealing. This is attributed to the reduction in the distortion of the lattice due to recovery. The occurrence of recovery prior to recrystallization at 1073 K in this type of material has been reported.^[17] Between 10 and 50 h of annealing, the variation in the velocity with time exhibits a sharp and continuous decrease. The sharp and continuous decrease is attributed to the decrease in the intensity of (111) reflections as observed in the x-ray diffraction (Fig. 1) due to the occurrence of recrystallization. Figure 6 shows the microstructures of the annealed specimens at 1073 K. The appearance of annealing twins in the microstructure (Fig. 6b) after 50 h of annealing is evidence for recrystallization. Coarsening of precipitates seen in the microstructures (Fig. 6b-d) is evidence for the progress of recrystallization from 50 h of annealing to 500 h of annealing. At 1123 K, the variation in the velocity with time exhibits a sharp decrease at 1 h of annealing followed by saturation at 5 h of annealing, indicating that recrystallization initiates and gets completed between 1 and 5 h of annealing. The microstructures of the annealed specimens at 1123 K are shown in Fig. 7. The microstructure after 1 h of annealing show no recrystallization while the microstructures after 10 and 100 h of annealing show completely recrystallized microstructures. Velocity measurements could distinguish clearly the recovery and recrystallization domains during annealing, and, hence, it will enable the determination of exact onset time for recrystallization.

3.2 Comparison with Hardness Measurements

Figure 8(a-d) compares the characterization of annealing by velocity measurements with that of the hardness measurements. At 923 K (Fig. 8a), hardness continues to decrease with annealing time due to recovery while velocity values remain almost constant. At 1023 K (Fig. 8b), hardness decreased initially with increasing annealing time. Between 1 and 50 h of annealing, there is no variation in hardness with annealing time except for a small peak. This is due to retardation of recrystallization by TiC precipitation. Beyond 50 h, hardness de-

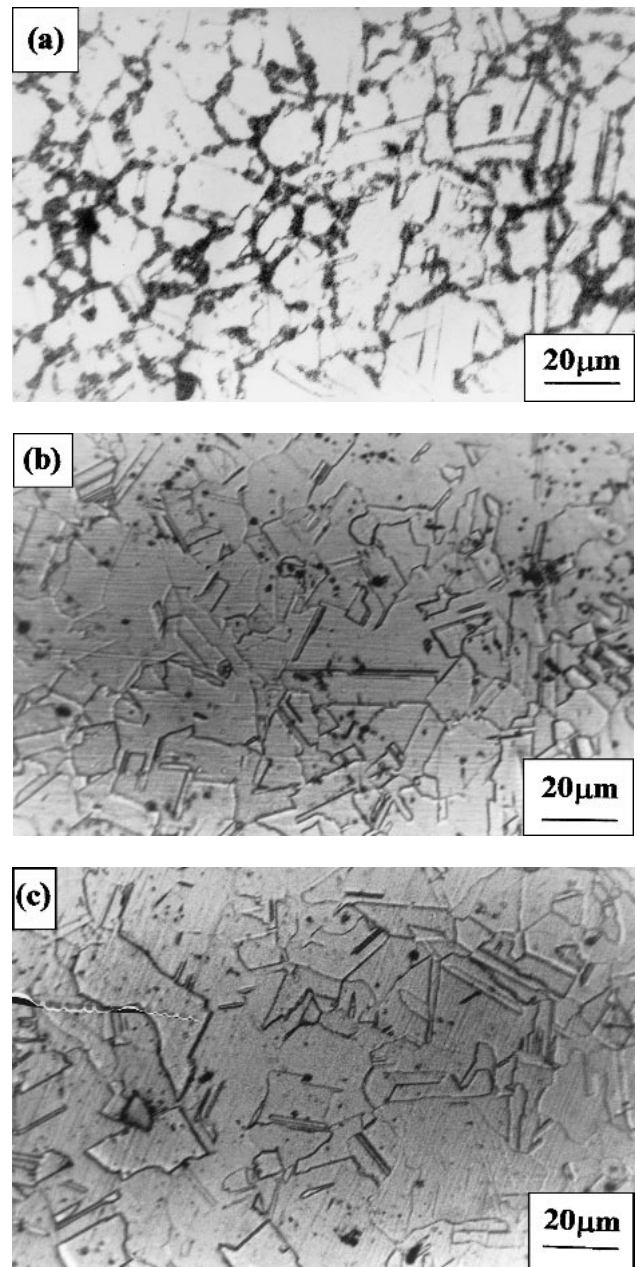


Fig. 7 Optical microstructures of the specimens annealed at 1123 K for times (a) 1 h, (b) 10 h, and (c) 100 h

creased with annealing time. Variation in velocity with annealing time exhibited small fluctuations between 1 and 100 h of annealing and the velocity values were the same or more than the velocity value in the cold worked condition. Beyond 100 h of annealing, velocity values decreased with increasing annealing time, indicating the occurrence of recrystallization. At 1073 K (Fig. 8c), hardness values decreased with annealing time right from 0.5 h of annealing and reached saturation at 500 h of annealing. A sharp decrease in the velocity values between 10 and 50 h of annealing indicates the onset of recrystallization. This was verified by estimating the volume fraction recrystallized using quantitative metallographic technique. The values

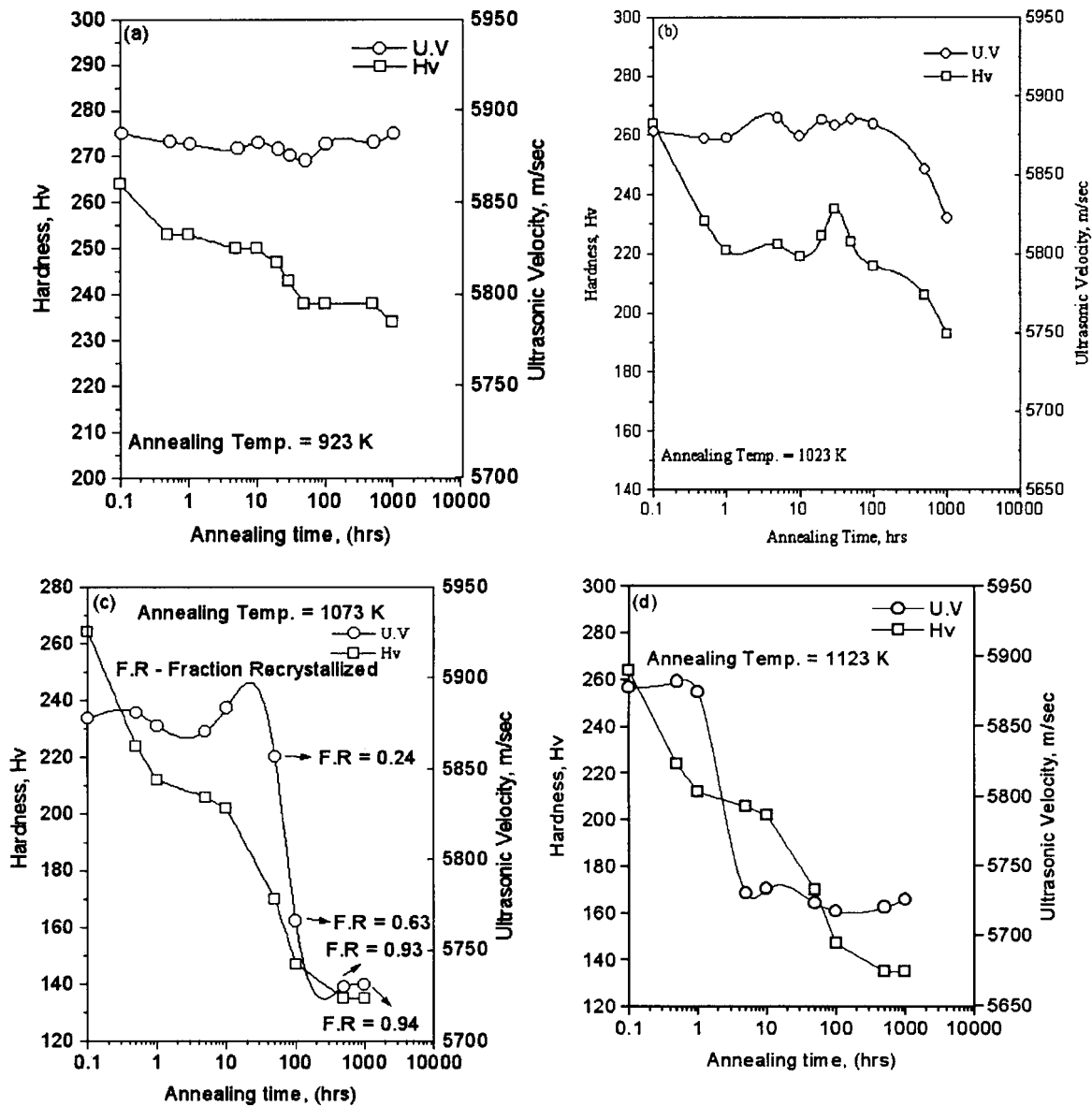


Fig. 8 Comparison of hardness measurements with velocity (20 MHz) measurements during isothermal annealing at (a) 923 K, (b) 1023 K, (c) 1073 K, and (d) 1123 K

are incorporated in the figure. After 10 h of annealing, volume-percent recrystallized was zero while 24% was volume-percent recrystallized after 50 h of annealing and 63% was volume-percent recrystallized after 100 h of annealing. After 1000 h of annealing, volume-fraction recrystallized reached 94%. Thus, onset of recrystallization occurs between 10 and 50 h of annealing at 1073 K. At 1123 K (Fig. 8d), velocity exhibited a sharp decrease at 1 h of annealing, indicating the onset of recrystallization. As velocity is sensitive to the change in texture due to recrystallization, it could predict the exact time for onset of recrystallization though precipitation occurs along with recrystallization. This is not possible using hardness measurements as the hardness decreased continuously from that of the cold worked value and reached a constant value after 500 h of annealing.

3.3 Effect of Frequency on the Characterization of Annealing Behavior

Figure 9(a-d) compares the variation of velocity with annealing time at different frequencies (2, 10, and 20 MHz). At 923 K, velocity measured using a 2 MHz probe increased with annealing time. During annealing at 923 K, only recovery and precipitation of TiC takes place. It implies that velocity measured at low frequency is only sensitive to the reduction in the distortion of the lattice due to recovery and is not influenced by TiC precipitates. The velocity measured using the 10 and 20 MHz probes remained almost constant with increasing annealing time. At high frequencies ultrasonic waves are expected to interact with precipitates, and that interaction could possibly suppress the increase in velocity caused by recovery. This is in

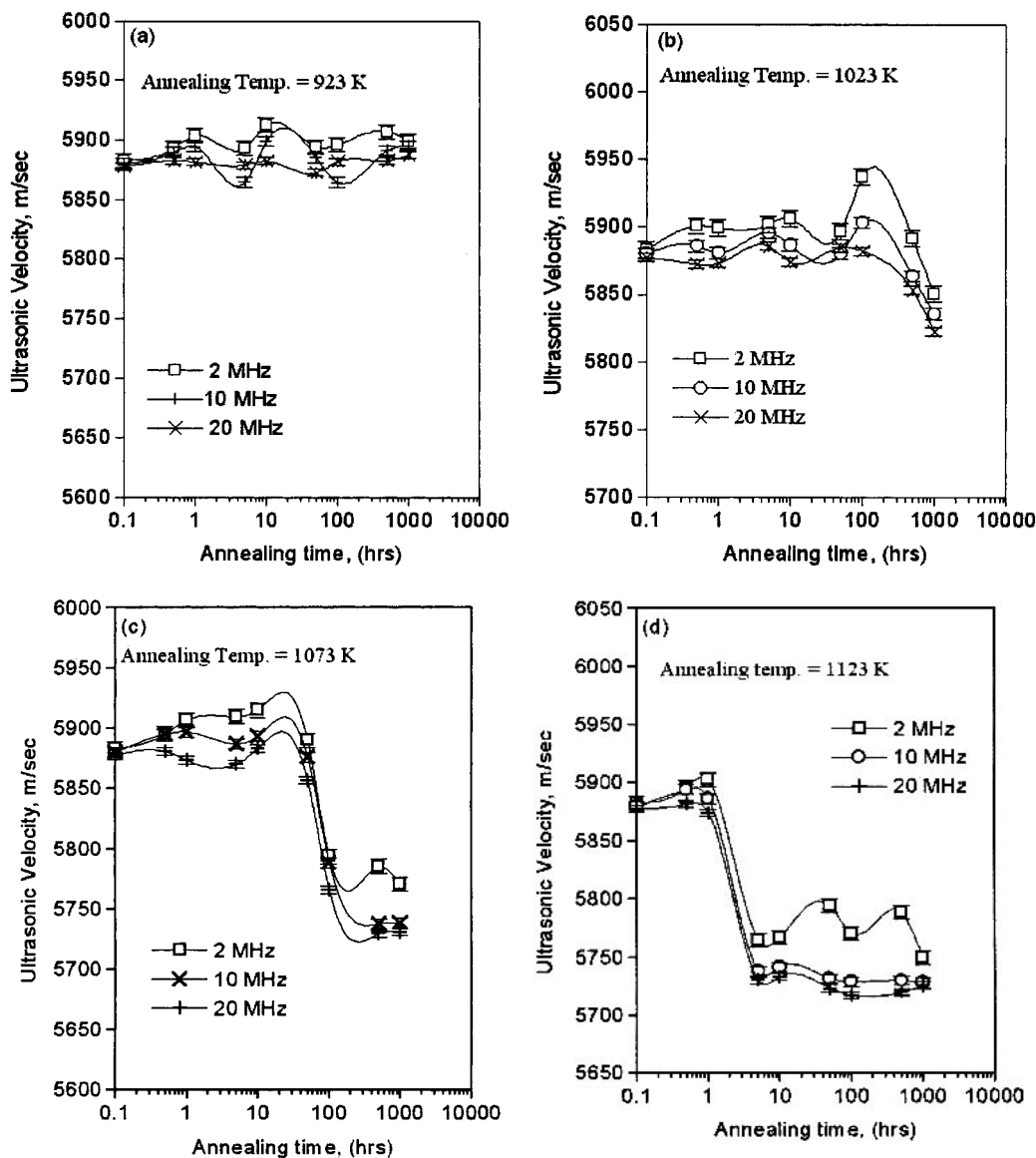


Fig. 9 Influence of ultrasonic frequency on the velocity measurements during isothermal annealing at (a) 923 K, (b) 1023 K, (c) 1073 K, and (d) 1123 K

agreement with observations that ultrasonic velocity measurements at high frequencies are only sensitive to study precipitation.^[22] At temperatures 1023, 1073, and 1123 K, velocity measured at all the three frequencies 2, 10, and 20 MHz on the isothermally annealed specimens showed the same onset and completion time for recrystallization. In the frequency range studied, the characterization of annealing behavior using velocity measurements is independent of the frequency of the ultrasonic waves.

3.4 Isochronal Annealing Behavior

Figure 10 shows the variation of ultrasonic velocity (2 MHz) with annealing temperature for the austenitic stainless steel isochronally annealed at 1, 100, and 1000 h. After 1 h of annealing, the velocity values for the samples annealed in the

temperature range 873-1123 K were greater at least by 50 m/s compared to that of the cold worked condition, while the velocity value for the sample annealed at 1173 K was lower by 150 m/s compared to that of the cold worked condition. Microstructural observations revealed the occurrence of recrystallization only in the sample annealed at 1173 K. The increase in the velocity observed in the samples annealed in the temperature range 873-1123 K was attributed to recovery. During recovery, annihilation of point defects takes place, which reduces the distortion in the lattice caused by cold work and also alters the modulus. Hence, there was an increase in velocity due to recovery. The decrease in velocity at 1173 K after 1 h of annealing was attributed to the decrease in the intensity of (111) reflection caused by recrystallization. After 100 and 1000 h of annealing, the variation of velocity exhibited an increase of 50-100 m/s in the temperature range 873-923 K followed by

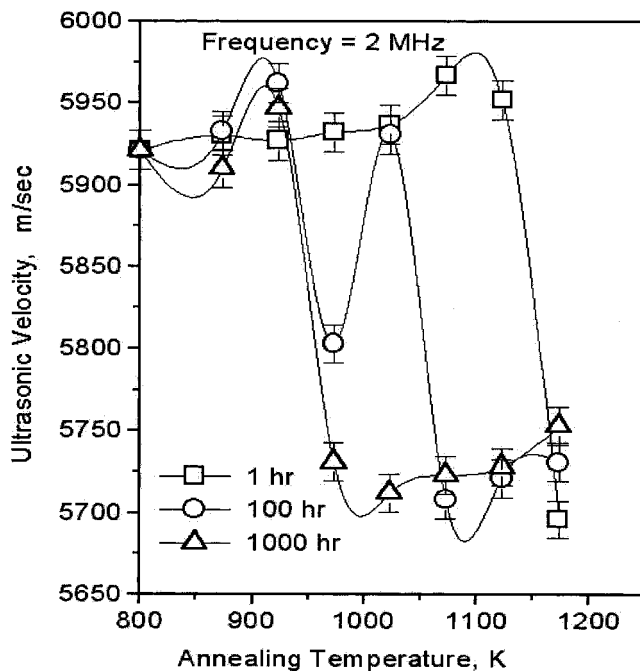


Fig. 10 Velocity as a function of annealing temperature during isochronal annealing

a drop at 973 K and a distinct peak at 1023 K, and continued to decrease until it reached saturation at 1123 K. Though precipitation interfered with recovery and recrystallization, the ultrasonic velocity being sensitive to textural changes predicted the onset temperature of recrystallization more precisely.

3.5 Correlation with Yield Strength

Figure 11(a-c) shows the comparison of variation of yield strength and ultrasonic velocity as a function of annealing temperature for different isochronal annealing times of 1, 100, and 1000 h, respectively. After 1 h of annealing, a continuous decrease in yield strength was observed in the temperature range 873-1123 K followed by a rapid decrease at 1173 K. The velocity values were higher in the temperature range 873-1123 K than the cold worked condition, followed by a rapid decrease at 1173 K. After 1 h of annealing, recovery has occurred in the temperature range 873-1123 K while recrystallization occurred at 1173 K. There was one to one correspondence between the variation in the yield strength and the velocity with annealing temperature in distinguishing the recovery and recrystallization regimes after 1 h of isochronal annealing. After 100 h of annealing, the yield strength decreased by about 200 MPa at 973 K followed by a peak at 1023 K, and then decreased continuously until it reached saturation at 1123 K. From the observation of yield strength, it appears that recrystallization has progressed considerably at 973 K. However, microstructural observation and velocity measurements indicate that only the onset of recrystallization has occurred at 973 K. The considerable decrease in yield strength was attributed to the dissolution of TiC precipitates caused by the onset of recrystallization. The dissolution of these precipitates during recrystallization is in agreement with the results reported in the literature.^[17]

There was one to one correspondence between the variation in the yield strength and the velocity with temperature in distinguishing the recovery and recrystallization regimes after 100 h of isochronal annealing, except at 973 K where the interaction between the TiC precipitation and recrystallization is maximum. After 1000 h of annealing, the variation in yield strength exhibited a sharp drop at 973 K and then reached saturation at 1023 K. Both yield strength and velocity predict recovery in the temperature range 873-923 K and complete recrystallization in the temperature range 973-1173 K. There was one to one correspondence between the variation in yield strength and the velocity as a function of annealing temperature for isochronal annealing at 1000 h. Thus, when the interaction between TiC precipitation and recrystallization is stronger, yield strength measurements were not accurate in assessing the extent of recrystallization. However, there was good correlation between the velocity measurements and the microstructural observations at all the temperature and time combinations used in the current study. Hence, when the interaction between the TiC precipitation and the recrystallization is maximum, velocity measurements were more accurate in characterizing the recrystallization behavior compared to yield strength measurements. Thus, mechanical property measurements are not as accurate as velocity measurements to study recrystallization behavior when there is secondary precipitation associated with recrystallization. We should use techniques such as ultrasonic velocity measurements which are sensitive to texture.

To study recrystallization kinetics, it is required to determine the onset time or time for 50% recrystallization. If a technique can exactly determine the onset time for recrystallization, then it will also facilitate the accurate determination of activation energy for recrystallization and, hence, the mechanism for recrystallization. We believe that ultrasonic velocity measurements is a useful technique in this regard. Also, using this technique, the exact recrystallization temperature for metallic alloys can be determined.

4. Conclusions

- 1) The variation of velocity with annealing time exhibited an increase in the recovery region at low frequency (2 MHz) while it remained almost constant at high frequencies (>10 MHz). At high frequencies (>10 MHz), the velocity did not exhibit an increase in the recovery region because of the enhanced interaction between ultrasonic waves and TiC precipitates. Velocity exhibited a sharp decrease in the recrystallization region due to the decrease in the intensity of (111) reflection (i.e., change in the texture). The velocity values reached saturation on completion of recrystallization, as there is no further decrease in the intensity of (111) reflection.
- 2) Ultrasonic velocity measurements could sense precisely the onset, progress, and completion of recrystallization, and, hence, the isothermal and isochronal annealing behavior of 15Cr-15Ni-2.2Mo-Ti-modified austenitic stainless steel was characterized more accurately by ultrasonic velocity measurements. The onset time (isothermal) or temperature (isochronal) for recrystallization were determined exactly.
- 3) The onset time or temperature for recrystallization was

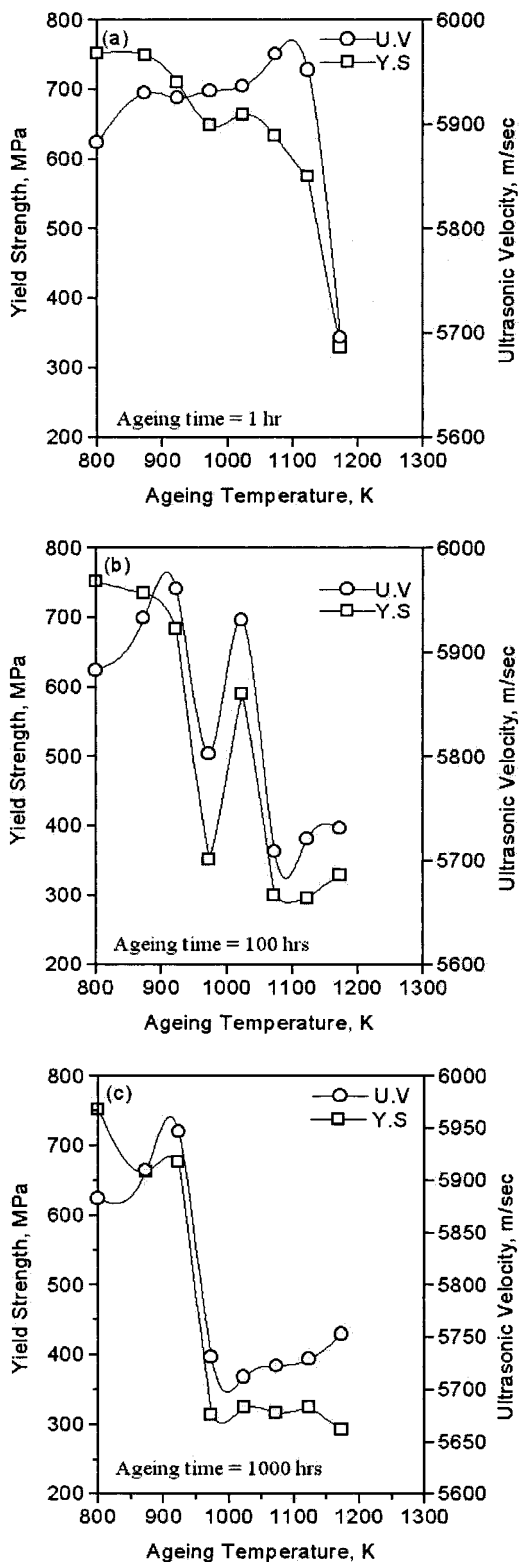


Fig. 11 Comparison of yield strength measurements with velocity (2 MHz) during isochronal annealing after (a) 1 h, (b) 100 h, and (c) 1000 h

found to be the same independent of the frequency (2, 10, and 20 MHz) of the ultrasonic waves used for the velocity measurements.

- Characterization of annealing behavior of cold worked Ti-modified austenitic stainless steel by velocity measurements is more accurate as compared to hardness and strength measurements. Velocity measurements is more suitable for studying recrystallization behaviors in materials where recrystallization is accompanied by secondary precipitation.

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