STUDY OF SPINODAL DECOMPOSITION OF A DUPLEX STAINLESS STEEL BY NON-DESTRUCTIVE TEST

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Abstract. *The initial stage of decomposition of the a phase in the duplex stainless steel in the temperature of 475oC is characterized for the mechanism of spinodal decomposition. This mechanism is responsible by the hardening of these steels. In this temperature, we have the spontaneous decomposition of the a phase by the formation of finely dispersed precipitates of a' phase in the matrix, which is responsible by the embrittlement at 475oC.In the present work, the decomposition kinetic at the temperatures 425oC, 475oC and 525oC was studied with the objective of developing non-destructive procedures to evaluate the embrittlement. Different times of treatment were applied at these temperature and the specimens were submitted to measure of sound velocity, impact test, hardness test and determination of geometry fractal of the fracture surfaces. The results, in temperature range of 425oC to 525oC, showed a correlation among values of sound velocity, absolved energy by impact and geometry fractal of the fractures, so we can notice that the mechanism of spinodal decomposition, which is characterized by the formation of modulate structure of a' phase, change the values of sound velocity, pointing as a non destructive parameter for follow the embrittlement kinetics.*

Keywords*: espinodal decomposition, non- destructive test, duplex stainless steel.*

1. Introduction

The spinodal decomposition is characterized by the spontaneous transformation of the matrix in material with miscibility gap. It forms a modulated structure of rich and poor composition. The riches regions are coherent with the matrix and lose their one at the end of the transformation forming a new phase. The modulated structure formed is responsible by the embrittlement of the material (Choo 1997 and Hedin 1996).

Duplex stainless steels have been used for many decades due to its excellent properties of mechanical strength and corrosion. These steels were introduced in the market during the thirties and it has been happened an accentuated development and use mainly during the last fifteen years. Today, the duplex stainless steels are in direct competition with the ferrite and austenitc stainless steels (Reick 1998).

The duplex stainless steel contains a mixture of ferrite and austenite grains. A microstructure of completely ferrite is formed during the solidification. The partial transformation to austenite happens during the cooling to produce the precipitate of austenite in the main ferrite (Hedin 1996). Due to its high mechanical and corrosion resistance, duplex stainless steels are widely used in systems of nuclear industries, as well as in others where they are exposed to severe environment conditons such as in the cellulose and petroleum industries. The superior property of these steels result mainly of the presence of the ferrite phase in the duplex structure. On the other hand, it is known that ferrite stainless steels become brittle due to the precipitation of the α' phase when heated in the temperature range between 300 and 500^{o}C for a long time (Kim 1997). This embrittlement in low temperature is known as 475^{o}C embrittlement which happens with a more accelerated precipitation kinetics. Recent works objecting the study on this embrittlement process indicates that the main mechanism responsible for it is the spinodal decomposition (Kim 1997).

The objective of this study is to verify the potential application of ultrasonic techniques to characterize thermal embrittlement of duplex stainless steels.

2. Experimental methods

The material used was an UNS-S 31803 duplex stainless steel with the following composition: C- 0.018, Si-0.45, Mn-1.48, P-0.002, S-0.001, Cr-22.22, Ni- 5.59, Mo- 3.080 and Fe- balance. Specimens were cut from a 12mm-thickness plate and subjected to thermal aging at the temperatures 425^{o}C, 475^{o}C and 525^{o}C for different holding time (12 to 175 hours) in order to obtain different degrees of hardening. A normal transducer of 2MHz was used for ultrasonic testing. The measurements of the sound velocities were performed by a digital signal processing method based on a pulse echo method. Charpy impact tests were made in standard specimens of 10x10x55mm. The microstructure and fracture surface were analyzed by scanning electronic microscopy. Fractal dimensions were obtained by the island method applied to fracture surface. Hardness values were obtained from Brinell testing. All the measurements were obtained with 5% of variation.

3. Results e discussion

Figure 1a shows the variation of Brinell hardness and Charpy impact energy for specimens aged at 475°C for different times. It is can be noticed that the hardness of the material increases rapidly for times up to 50 h, and at a smaller rate between 50 h and 100 h until it stabilizes for time superior to 100 h. Charpy energy results decrease up to 50 h and after that they remain constant. The rapid increase on hardness in the early stage of aging is related to the phenomenon of spinodal decomposition within the ferrite phase, which form a modulated structure of chromium rich regions alternated with Cr poor ones, that precedes the formation of a α' phase rich in chromium. The microstructure modulation and compositional alteration in the ferrite phase act as a strong barrier to dislocation motion increasing the hardness of the material. The spinodal decomposition phenomenon has been studied in others system, (Choo 1997) such as FeMnAl alloys and it was reported that the hardening phenomenon is divided in two stages: firstly, it occurs the spinodal decomposition and secondly, it occurs by elastic stress; this stress is generated by distortions in the matrix due to the growth of the α' phase in the Cr-enriched regions. From figure 2a , it can be considered that for times up to 25 h the mechanism responsible for hardening would be the spinodal decomposition and for times above 50 h it would be the formation of the α' phase and its growth. The second stage would be responsible for the poor values of impact energy above 50 hours of aging.

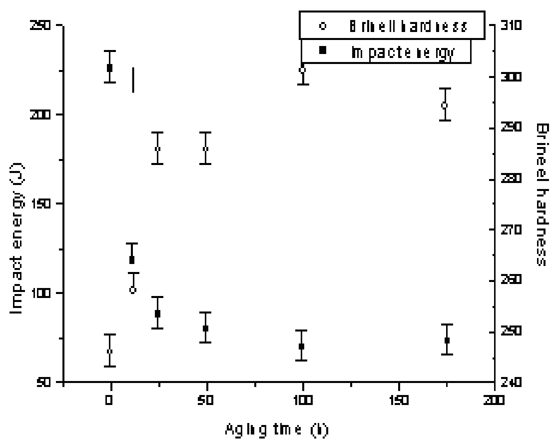

Figure 1a

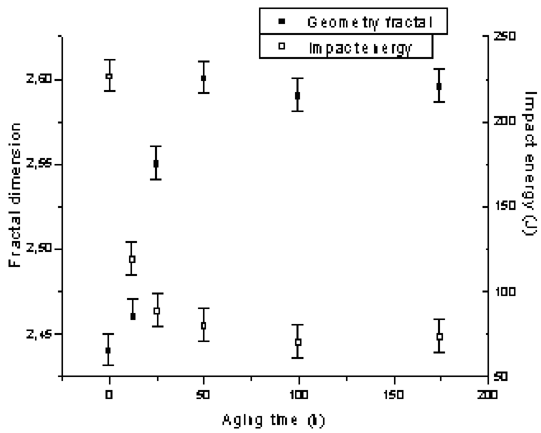

Figure 1b

Figure 1. a) Variation of the Brinell hardness and the impact energy against the time of aging at 475°C. b) Variation of the fractal dimension and the impact energy with the aging time at 475°C.

The analysis of fracture surfaces showed a ductile fracture for samples in the as-received condition (solution-treated). For specimens aged at 475°C, a brittle fracture was observed for times above 50 h. The fracture surfaces were analyzed by the method of the islands. By this method, one can obtain values of the surface fractal dimension for each aging time. It was objected to analyze the existence of a correlation between the fracture mechanism and the fractal geometry. The results of the fractal dimension for different aging times and the values of impact energy are presented in the figure 2b. One can observe an increase of the values of fractal dimension for times up to 50 h following the same trend of impact energy results. Values of fractal dimension of 2,60 and 2,44 were found for brittle behavior and ductile mechanisms, respectively. The correlation between fractal dimension and fracture mechanism have been studied by different authors (Mandelbrot 1984, Mu 1988, Hildres 1999), showing that there is a controversy about the existence of a characteristic fractal dimension for the several fracture mechanisms. However, the present results showed a clear correlation between them.

During the last two decades, ultrasonic testing was developed as an efficient tool for materials characterization. By their passage through the material, acoustical waves carry out a multitude of information on the mechanical and physical properties of the material (Jianfeng 1996,Smith 2002, Badidi 2003). Taking into account the results of the figure 1b and that aging times above 50 h are already enough for the material embrittlement, the studies of the variation of the sound velocity were measured until 50 h. The objective was to determine non-destructive parameters capable to characterize the kinetics of spinodal decomposition of duplex stainless steels.

The values of sound velocity and fractal dimension are shown in the figure 2a. It can be noticed that the sound velocity decreased with the increased time. It must be remembered that the spinodal decomposition occurs in this stage. The sound velocity appears as a sensitive parameter to follow the spinodal decomposition.

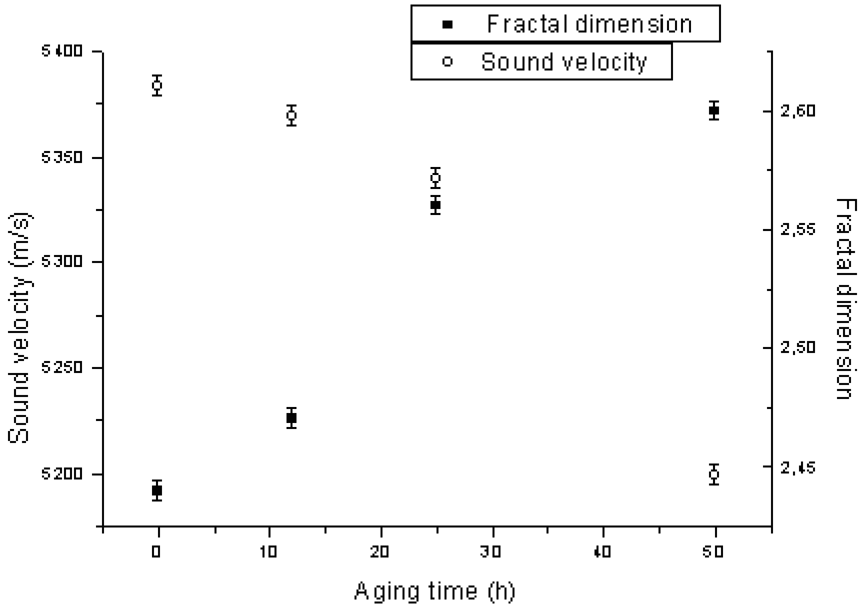

Figure 2. Variation of the sound velocity and fractal dimension for the specimens aged at 475^{o}C.

In the early stage of spinodal decomposition, there is the formation of a modulated structure of Cr-rich and Cr-poor regions. The amplitude of the structure increases with the aging time. The composition fluctuation caused will act as barriers to the sound propagation in the material and the sound velocity will decrease. The ultrasonic detection of thermal embrittlement of duplex stainless steel was studied by Shigeyuki (1996). The enbrittlement was interpreted in terms of the decrease in dislocation mobility by the modulated structure due thermal aging. They showed a different variation in sound velocity. It was found an increase of it with the aging time but they were not able to explain the reason.

The figure 3 shows the variation of the sound velocity in the temperature range of 425^{o}C to 525^{o}C. It can be noticed that the sound velocity decreases as the aging time increases. The kinetic of spinodal decomposition grows in function of the temperature according to the increase of the atoms mobility, so the modulated structure formed acts as barrier to the sound mobility. The results show that the sound velocity seems to be an effective parameter for characterization of the embrittlement in the initial stage of decomposition of the ferrite phase.

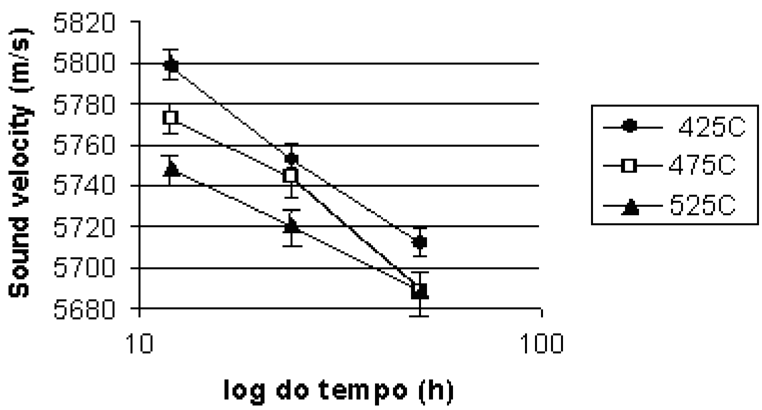

Figure 3.Variation of the sound velocity in the temperature range of 425^{o}C to 525^{o}C.

4. Conclusions

The variation of the sound velocity was studied in a duplex stainless steel with the objective of evaluating non-destructive parameters to follow the kinetics of transformation caused by spinodal decomposition. Besides, the fractal dimension of the fracture surface was determined and correlated with those values. The following conclusions can be drawn:

1. Values of fractal dimension showed a correlation between the fracture mechanism and the fractal theory where there were controversies in the literature. Values of fractal dimension of 2,60 and 2,44 were found for brittle and ductile mechanisms, respectively.
2. The sound velocity proves to be effective to follow the kinetics of spinodal decomposition in duplex stainless steel. However, farther studies with others parameters as coefficient of sound attenuation is need in order to have more information of the influence of the modulated structure caused by spinodal decomposition over sound velocity.

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