Characterization of the states of aging of HP austenitic stainless steels through spectral analysis of ultrasonic signals

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Abstract. The ultrasonic non-destructive evaluation is a valuable tool for materials characterization in the industry. It presents advantages such as high speed of testing, low operational cost and portability for field inspection. The great majority of engineering materials present microstructural heterogeneities such as pores, inclusions, second phase particles and precipitates, which may change their response to ultrasound. The Fast Fourier Transform is useful for converting signals from the time domain to the frequency domain in order to reveal information that is not evident in typical ultrasonic signals. The Fast Fourier Transform (FFT) is an important signal processing algorithms to obtain the Fourier Transform; its application in ultrasonic signals is already well established in the literature and has been subject of studies by several authors. Spectral analysis using the FFT of back wall echoes may allow the microstructural characterization of a material.

The present study aims at the characterization of the aging condition of centrifugally cast heat-resistant HP stainless steels through the post-processing of ultrasonic signals. This material has been widely applied at the oil industry in reformer and pyrolysis furnaces tubes due to its high creep resistance and high mechanical performance while exposed to severe operational conditions. However, these conditions can cause microstructural changes in HP steels and may reduce their creep and mechanical resistance, leading the material to failure. Therefore, in order to ensure the reliability of these structures, the development of non-destructive techniques capable of monitoring the states of aging this steel is required. The FFT was applied on the ultrasonic signals obtained from three HP steel samples, each one presenting different states of aging. Spectral analysis was conducted in back wall echo of these specimens. The results indicate that it is possible to characterize different states of aging in HP steel samples through the proposed methodology.

1 Introduction

Characterization of materials using non-destructive ultrasound technique is a promising tool in the Oil and Gas industry. Ultrasonic signal processing using Fast Fourier Transform (FFT) allows obtaining information on the characteristics of the material [1].

HP heat resistant, austenitic stainless steel tubes are obtained by centrifugal casting process. This steel has been used in the Oil and Gas industry in the manufacture of tubes used in reformer and pyrolysis furnaces. This type of furnace operates in an intermittent way and is submitted to high temperatures and pressures. Inside the reformer furnaces, the tubes are placed in vertical harps and the burners normally placed on the roof, supplying energy to the steam hydrocarbon gas mixture that circulates inside the tubes, and acting as heat exchangers. These alloys are used because of their characteristics of high resistance to oxidation at high temperatures, thermal stability, resistance to creep and competitive cost [2,3].

HP steel has an As Cast microstructure formed by austenitic dendrites decorated a complex eutetic carbide network. These steels are stable up to an operating temperature of 650 °C, however, when temperature increases, there is an intradendritic precipitation of secondary Cr-rich $M_{23}C_6$ carbides. For niobium modified HP steels, at temperatures above 700 °C, it is observed the in situ transformation of the primary niobium carbide to a nickel–niobium, silicide, identified as the G-phase. Associated with the formation of G-phase, lenticular voids can be formed for long term service under constant load at elevated temperatures [2]. Theses voids lead to the formation of interdendritic creep cracks, which is the main failure in service for this type of furnace.

Preventive maintenance of reformer furnaces is necessary, because unscheduled stoppages generate large economic losses. In the current economic scenario, the strategic planning of Oil and Gas industries had to be revised from a positive point of view, this brought a new perception of non-destructive tests.

Ultrasound technique, coupled to signal processing is a tool acknowledged in literature to assess microstructural changes in materials [1]. The spectral analysis of the background echo signal of ultrasound signals has been an advantageous technique, because it can be applied in materials with complex geometries. In addition, these analyses can evaluate microstructural elements in depth, which represents an incentive for the characterization of the material, object of this study [1,4].

2 Experimental Methods

2.1 Material

Three samples of HP steel with different aging stages were analyzed. The aging stages were designed as: Stage I, IV and V. The state of aging criterion is defined elsewhere [5]. The alloy chemical composition is presented in Tab. 1.

Tab. 1: Chemical composition of the HP-Nb alloy [6].

	C	Mn	Si	P	S	Mo	Cr	Ni	Nb	Ti
Composition [% wt]	0,45	0,91	1,24	0,021	0,010	0,031	27,0	34,6	0,74	0,05

The samples were removed from a reformer tube that was in operation during 70000 hours and exposed to different service temperatures. Each sample was removed from different heights based column. The thickness of the HP steel samples are: 11,61 mm to Stage I, 11, 53 mm to Stage IV and 11,40 mm to Stage V.

2.2 Metallographic analysis

Surface preparation for the scanning electron microscopy (SEM) analysis followed the following steps: the samples were removed from the tube cross-section and sanded with 100, 200, 320, 400, 600 up to 1500 grain size sandpaper and each time the sandpaper was changed, the samples were rotated 90°. Samples were analized on the surface which corresponds to the cross section of the tubes. The microstructural characterization was performed using a ZEISS scanning electron microscope (SEM) model DSM 940, using non-etched specimens in the backscattered electron mode. Before the sectioned specimens were ground using 100-1200 grit sic paper.

2.3 Ultrasonic testing

The adopted ultrasound technique was pulse-echo, using contact mode and honey coupling and a ISONIC 2005 ultrasound equipment with a 1,6 MHz (Imasonic®) non-focused longitudinal wave transducer, shown in Fig. 1. The signals acquired were digitized through a Tektronix oscilloscope, model MSO 4034 with 250 MA/s sampling frequency.

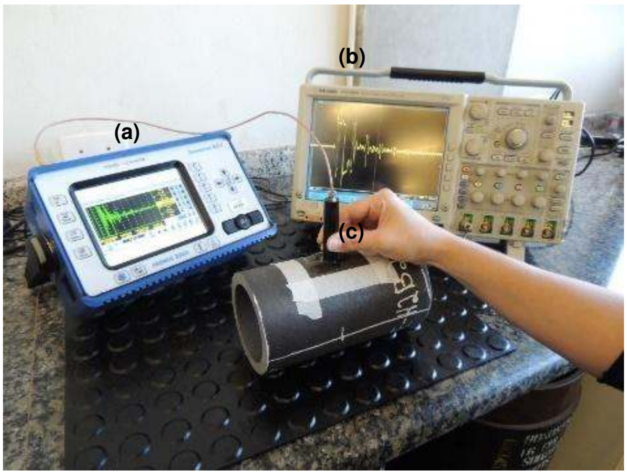


Fig. 1: Experimental setup for ultrasound signal (a) ultrasonic testing equipment, (b) transducer and (c) oscilloscope.

2.4 Ultrasonic signal processing

After ultrasound signal acquisition, the data were exported to a routine developed in Matlab® environment. Initially processing performed was the cross-correlation of the 30 A-scan signals acquired, aiming at correcting the phase difference among signals. Then, the mean of the 30 signals was calculated to obtain a single A-scan signal for each aging stage.

The identification of the background echoes in the A-scan signal is of fundamental importance for the subsequent analyses. Fig. 2 shows the location of the background echoes for one of the analyzed samples.

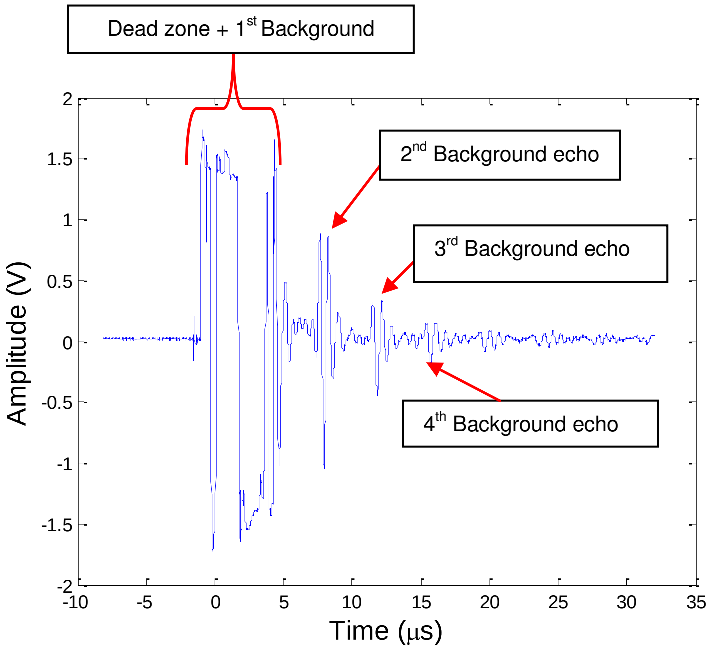


Fig. 2: A-scan signal with location of background echoes of the sample in Stage I.

In the A-scan signal, the stretch of the second background echo was windowed with at least 512 points, for subsequent application of FFT (Fast Fourier Transform). Thereafter, the constant level (known as dc level) of this stretch was removed and the computation calculation of DFT was carried out through a high performance algorithm known as FFT, obtaining the background echo spectra for each aging stage.

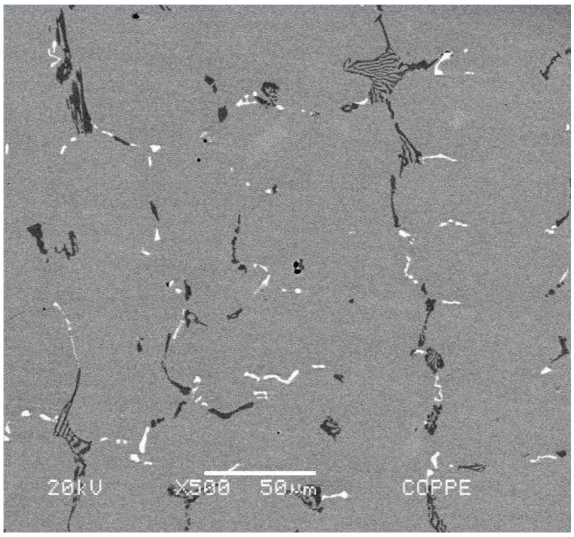
3 Results

3.1 Quantitative Microstructure characterization by SEM

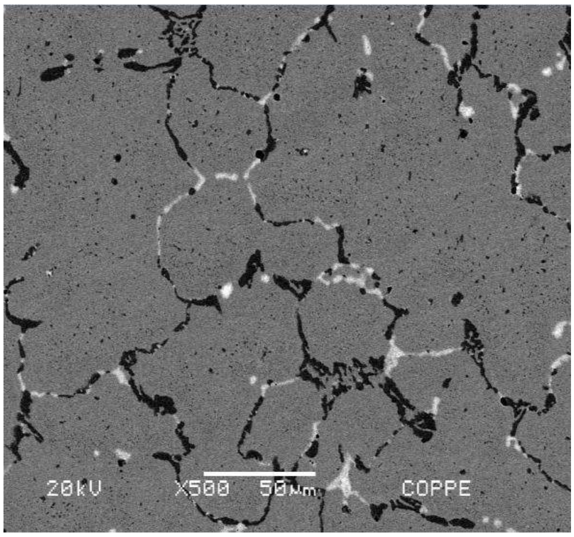
According to Fig. 3 (a), the microstructure of the sample with aging Stage I is composed by the austenitic matrix and coalesced interdendritic chromium rich carbide ($Cr_{23}C_6$) and niobium carbide (NbC), with some $M_{23}C_6$ secondary carbides finely dispersed in the matrix. The eutectic interdendritic carbides form films outlining the dendritic structure, as indicated in Fig. 3 (a), chromium (dark color) and niobium (white color) carbide.

In the aging Stage IV, shown in Fig. 3 (b), the primary interdendritic Cr-rich carbide coarsened significantly during service exposure, in contrast, the NbC remained approximately the same size as in the Stage I. A uniform and intense precipitation of secondary carbides of the type $M_{23}C_6$ is formed in the matrix increasing the carbides volume fraction.

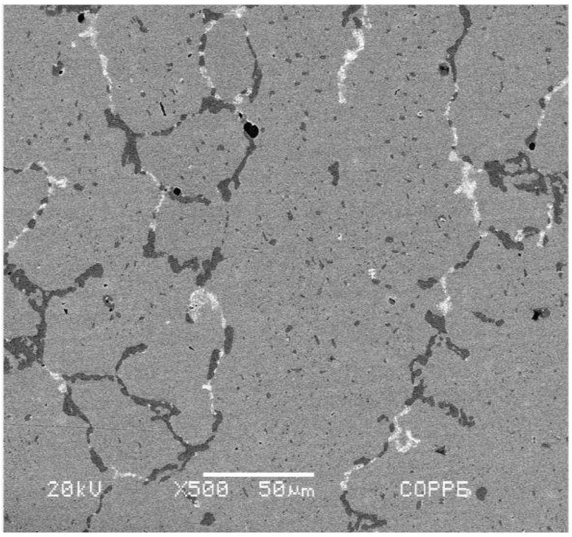
When the material is exposed to higher temperatures, when the sample with aging Stage V, shown in Fig. 3 (c), it presents larger coalesced secondary chromium carbides in the intradendritic region and the NbC is partially transformed into phase G.



(a)



(b)



(c)

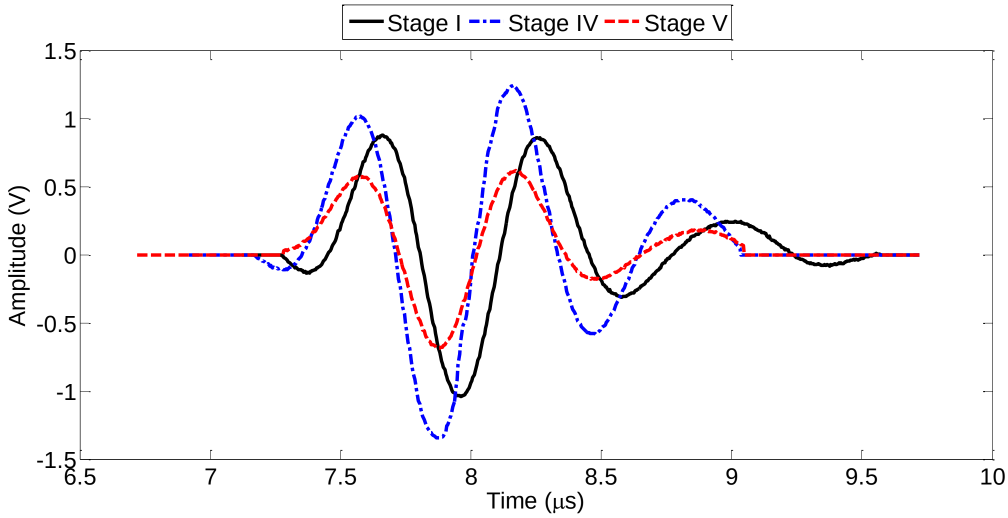
Fig. 3: Micrograph obtained in SEM with the microstructures of the three aging conditions (a) Stage I, (b) Stage IV and (c) Stage V, for the samples of HP stainless steel.

3.2 Ultrasonic spectral analysis for characterization

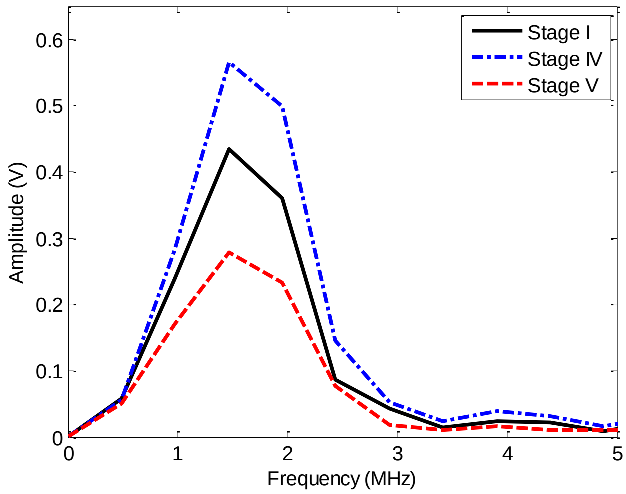
The background echoes of the ultrasound signals were analyzed in the time domain for the three aging stages shown in Fig. 4 (a). It can be observed that the amplitude of the ultrasound signal is influenced by the microstructural condition of the material. Stage IV has greater amplitude than the other stages, Stage I has an intermediate amplitude and Stage V has the smallest amplitude, as shown in Fig. 4 (a).

By transforming the signal in the time-domain into the frequency domain through FFT, in the region of the A-scan signal of the background echo, we observe the same performance in terms of amplitude, as shown in Fig. 4 (b). The amplitude is greater for Stage IV, intermediary for Stage I and smaller for Stage V. There is also prioritization of low frequencies in the frequency domain, next to the transducer central frequency.

Relating to both results of the ultrasonic and the microstructural characterization, we can conclude that the Stage IV has greater amount of precipitates dispersed in the matrix than Stage V, and those precipitates reduced the average free path for ultrasonic wave propagation, which was reflected in the amplitude of the background echo spectrum.



(a)



(b)

Fig. 4: Ultrasound signal of the three aging stages, I, IV and V of an HP steel (a) in the time domain and (b) in the frequency domain.

4 Conclusion

The analyses of the background echo of ultrasound signals for the samples of HP steel showed that there is influence of the aging conditions. Both background echo amplitudes, in the time domain and frequency domain, presented the same performance: greater amplitudes for Stage IV, intermediary for stage I and smaller for Stage V. Our results proved the potential of the technique for the characterization of the three aging stages of HP steel.

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6 References

[1] KRÜGER, S. E., J. M. A. Rebello, and P. C. De Camargo. "Hydrogen damage detection by ultrasonic spectral analysis." NDT & E International 32.5 (1999): 275-281.
[2] DE ALMEIDA, L.H. et al, Microstructural characterization of modified 25Cr–35Ni centrifugally cast steel furnace tubes. s.l.: ELSEVIER, Materials Characterization, Vol. 49, pp. 219-229, 2003.
[3] ASM, Metals Handbook: Vol. 1: Properties and Selection: Irons, Steels and High-Performance Alloys. 10 ed. USA, 1990.
[4] VARY A. Material property characterization. In: Nondestructive testing handbook, vol. 7 2nd ed. USA: ASNT, 1991: 384-431.
[5] SILVEIRA, T.L. da, LE MAY, I. . Reformer furnaces: materials, damage mechanisms and assessment. The Arabian Journal for Science and Engineering, 2006, Vol. 31, pp. 99-119.
[6] SIQUEIRA, N. C. de, Caracterização do Estado de Envelhecimento de um Aço HP por Ensaio Não Destrutivo de Ultrassom. M.Sc. UFRJ/COPPE, Rio de Janeiro, RJ, Brazil. 2016.