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# Anomalous ultrasonic attenuation in ferritic steels at elevated temperatures

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## 1. Experimental

37 The present work was carried out as part of a pan-European 38 research project into the control of microstructures in steel during 39 heating and hot working where laser-ultrasonics (LUS) have been 40 used to investigate the microstructural state of steels at elevated 41 temperatures [1]. The virtue of LUS is that the instrumentation is 42 remote from the material being studied so is uniquely suited for high temperature metallurgical investigations of this sort [2]. Here 43 44 we report on two steels which were supplied in the hot rolled and coiled condition. Steel DP600 is intended for processing as a dual-45 phase structure while S550 is a titanium/niobium-microalloyed 46 high strength low alloy (HSLA) steel. Their chemical compositions 47 are given in Table 1. Annealing experiments were carried out in a 48 Gleeble machine by heating at a constant rate of 5 °C/s to temper-49 atures of 950 °C, 1050 °C or 1150 °C and then holding at these 50 temperatures for periods of time up to 10 min. No deformation 51 was applied in these experiments. 52

A laser-ultrasonic system coupled with the Gleeble machine was used, acquiring ultrasonic signals during annealing experiments at a rate of 10 Hz to capture rapid microstructure variations. Measurements were performed in reflection mode with a laser generation spot size of 2 mm and detection spot size of about

http://dx.doi.org/10.1016/j.ultras.2016.03.005 0041-624X/© 2016 Published by Elsevier B.V. ABSTRACT

An unexpected peak in attenuation has been observed at  $\sim$ 800 °C when heating low carbon steels in a laser-ultrasonic instrument. An explanation is given in terms of enhanced crystalline anisotropy with increasing temperature in the bcc ferrite range combined with subsequent transformation to austenite at still higher temperatures. An analysis based on theoretical models of attenuation in the Rayleigh regime is in good agreement with the experimental observations.

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3 mm. To generate the ultrasonic wave, a short (5 ns), energetic (150 mJ), green (532 nm) light pulse from a frequency-doubled Nd:YAG laser was employed. The laser interferometer employed a longer-pulse Nd:YAG laser operating at 1064 nm (infrared), with a pulse duration of 50  $\mu$ s and a pulse energy of approximately 70 mJ. A confocal Fabry–Perot interferometer was employed operating in reflection and with a detection bandwidth of 75 MHz. The measurements applied to longitudinal *P* waves. More details about the experimental set-up have been given elsewhere [3].

The basic equation used to interpret ultrasonic attenuation measurements of longitudinal waves is:

$$\alpha(f) = a + bf^3 \tag{1}$$

where the first term is related to ultrasound absorption (considered independent of frequency) and the second term is related to scattering. The *a* and *b* values are treated as fitting parameters where *a* is strongly temperature dependent and is assumed independent of grain size. In the measurements, a single-echo approach is used in which the attenuation is determined using the Fourier components of an echo A(f) and those of a reference *Aref* (f), with the relation:

$$\alpha(f) = \frac{1}{2e} \left( 20 \log_{10} \left( \frac{Aref(f)}{A(f)} \right) - C \right)$$
(2)

Here, *C* is an offset (positive or negative) for the amplitude variations (in dB) from one laser shot to another. The reference signal is taken at room temperature in the beginning of the experiment.

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#### Table 1

Chemical compositions of the steels in this investigation (wt%).

Steel	С	Mn	Ti	Nb
DP600	0.11	1.90	_	-
S550	0.07	0.95	0.055	0.060

Such reference signal of low attenuation in same conditions is used
to remove the diffraction effect in the calculation of attenuation at
high temperature. The ultrasonic frequency content used for attenuation measurements was typically from 2 to 25 MHz while the
actual attenuation values given below were evaluated at 20 MHz.

Results from the laser-ultrasonics were complemented with
some dilatometer measurements to determine independently the
ferrite to austenite phase transformation on heating. These were
carried out in a Bähr dilatometer. The same heating rate of 5 °C/s
was adopted in this case. The initial microstructure of the steels
was checked using optical microscopy.

## 97 2. Results and discussion

98 Fig. 1 shows attenuation results as a function of time during the 99 in-situ annealing experiments with the temperature profile superimposed. The high temperature data, above 900 °C, are quite straightforward to understand on the basis that grain growth in the austenite causes an increase in the attenuation. Similar behaviour has been reported for numerous steel compositions as well as other metals such as copper e.g. [4–7]. At 950 °C, virtually no grain growth occurs in either steel and attenuation remains at a low and constant level, even after holding for the longest times of 10 min. At this temperature, particles of aluminium nitride (AlN) are known to be stable and these provide a Zener-pinning effect [8] inhibiting grain growth. At 1050 °C AlN particles are mainly dissolved so that grain growth takes place in the DP600 steel, increasing the attenuation. However, the S550 material contains, in addition, particles of niobium carbonitride (Nb(C,N)) that are still stable at 1050 °C and prevent grain growth in this steel. At 1150 °C, both steels show enhanced attenuation due to grain growth but it occurs more rapidly and to a greater extent in the DP600 case where no Nb(C.N) is present. Thus, the high temperature observations can be well rationalised on the basis of established metallurgical principles e.g. [9].

The unexpected phenomenon revealed in these experiments was the appearance of low temperature peaks that were quite reproducible and apparently corresponded with the steel still remaining in the ferritic (bcc) state. These peaks are asymmetrical,



Fig. 1. Attenuation data at 20 MHz measured during annealing of two steels including holding at three different peak temperatures. Heating profiles are shown as solid lines and the Curie temperature by dashed lines.

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