A NEW METHOD FOR THE MEASUREMENT OF ULTRASONIC ABSORPTION IN POLYCRYSTALLINE MATERIALS

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INTRODUCTION

In general, the ultrasonic attenuation in polycrystalline materials at room temperature (RT) is described in terms of scattering losses and absorption losses. Ultrasonic scattering is caused by the grain structure of the material whereas the interaction of the ultrasonic wave with lattice imperfections (e.g. dislocations, Bloch walls) leads to energy absorption. Usually, it is impossible to separate the different contributions by using conventional pulse-echo techniques which measure the total ultrasonic attenuation plus some artificial attenuation due to specimen geometry and sound-field divergence. Only if special assumptions on the frequency dependence of each contribution can be made, a separation might be possible /1/. Recently, several techniques have been proposed in order to measure ultrasonic absorption directly. One technique is based on resonance measurements in small cylindrical specimens by exciting standing waves at frequencies below 1.2 MHz /2/. A second technique uses the infrared detection of the heat produced by the ultrasonic absorption /3/, and a third technique is based on ultrasonic diffusion measurements /4/. This paper presents a new method for direct absorption measurements in polycrystalline materials by measuring ultrasonic reverberation. The method is especially appropriate in the case of large scattering contributions usually present in coarse grained steels.

DESCRIPTION OF THE METHOD

The block diagram of the experimental set-up is shown in Fig. 1a. The ultrasonic system used enables burst excitation of the ultrasonic probe, and a logarithmic amplification of the received signals. Ultrasonic pulses with a length of usually 10-20 cycles are insonified into the specimen using contact technique. Initially, the ultrasonic energy is contained within a volume \( V_p \) which is determined by the pulse length and the probe aperture. For the frequencies and the probes considered here, \( V_p \) is in the order of 1 cm\(^3\). After a time \( t_1 \), depending on the amount of scattering as well as on the volume \( V_e \) and the geometry of the specimen, the ultrasonic energy will be equally distributed within the whole specimen volume provided the specimen is not too large. From that moment onwards the amplitude decay of the received ultrasonic signals
exhibits a considerably smaller slope than for times below $t_1$ (see Fig. 1b). This new slope can be associated with the ultrasonic absorption coefficient $\alpha_A$ characterizing the absorption of ultrasonic energy by intrinsic material properties. The important point is that the ultrasonic scattering, which is necessary to reach the condition of homogeneous energy distribution, obeys the law of energy conservation and, thus, does not contribute to the amplitude decay for $t > t_1$. Obviously, the method cannot be applied to non-scattering (or weakly scattering) materials, because the ultrasonic energy would essentially remain in the initial pulse until complete absorption. The ultrasonic signals after $t > t_1$ may be considered as a kind of reverberation and, therefore, the method is called the reverberation method in what follows. It should be mentioned that the reverberation signal consists of a (unknown) mixture of longitudinal (L) waves, shear (T) waves and surface (R) waves due to the mode conversion by the scattering processes as well as by the reflections at the specimen boundaries. In fact, the reverberation signal can be picked up with any probe (L, T or R) of the same frequency at any specimen position. Because no definite propagation velocity can be attributed to the reverberation signals, all absorption coefficients measured with this method are given in units of dB/ms.

Obviously, the method is restricted with regard to the specimen volume. In the case of very small absorption coefficients a rough estimation yields for the upper limit of the specimen volume $V_S$

$$V_S = V_p \cdot \frac{D_0 (dB) - D_1 (dB)}{10^{20}} \quad (1)$$

Here, $D_0$ is the dynamic range of the initial ultrasonic signal, $D_1$ is the dynamic range necessary for signal evaluation, i.e. the determination of the slope of the logarithmic signal, and $V_p$ is the initial pulse volume as explained above. With $D_0 = 80$ dB, $D_1 = 20$ dB and $V_p = 1$ cm$^3$ one finds $V_S = 1000$ cm$^3$. Taking into account actual absorption coefficients and the reduced dynamic range of ultrasonic probes at higher frequencies, $V_S$ will be below 100 cm$^3$ in realistic cases. If the specimens are very small, losses due to mechanical damping by the ultrasonic probe have to be considered as can be seen from Fig. 2. Here, $\alpha_A$ was measured as a function of the specimen volume $V_S$ using cylindrical bars. The result is well described by the relation (solid line in Fig. 2):

$$\alpha'_A = \alpha_A + K/V_S \quad (2)$$

Here, $\alpha'_A$ is the measured absorption coefficient, $\alpha_A$ is the true absorption coefficient and $K$ depends on the ultrasonic probe. Concerning the specimens investigated in this work, $V_S$ was usually of the order of 10 cm$^3$.

Keeping in mind the limitations mentioned above, the method is very appropriate to measure absorption coefficients between 0 dB/ms and approximately 400 dB/ms. This range is usually encountered for most steels at room temperature (RT) and frequencies below 20 MHz. Under these conditions the ultrasonic absorption is mainly attributed to the interaction with dislocations /5,6/ and, in ferromagnetic materials also with the magnetic structure /7,8,9,10/. The reverberation method was used to investigate the influence of these parameters on the ultrasonic absorption. Some examples are demonstrated in what follows showing the high sensitivity of the method.
Fig. 1  
a) Experimental set-up for ultrasonic absorption measurements  
b) Example of measuring signal

Fig. 2  Influence of specimen volume on the absorption measurement as measured using cylindrical bars

Table 1. Microstructural and ultrasonic data of some investigated specimens. Ultrasonic frequency: 5 MHz (shear wave)

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Microstructure</th>
<th>Carbon content (wt%)</th>
<th>Grain size (µm)</th>
<th>α (db/ms)</th>
<th>α_A (db/ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ferrite</td>
<td>0.05</td>
<td>122</td>
<td>948</td>
<td>118.0</td>
</tr>
<tr>
<td>2</td>
<td>Ferrite/Pearlite</td>
<td>0.12</td>
<td>80</td>
<td>753</td>
<td>93.8</td>
</tr>
<tr>
<td>3</td>
<td>Ferrite/Pearlite</td>
<td>0.20</td>
<td>43</td>
<td>334</td>
<td>85.0</td>
</tr>
<tr>
<td>4</td>
<td>Pearlite</td>
<td>0.79</td>
<td>30</td>
<td>251</td>
<td>20.6</td>
</tr>
<tr>
<td>5</td>
<td>Austenite (Ni-base)</td>
<td>0.15</td>
<td>21</td>
<td>552</td>
<td>21.9</td>
</tr>
<tr>
<td>6</td>
<td>Austenite (Fe-base)</td>
<td>0.07</td>
<td>80</td>
<td>1240</td>
<td>10.0</td>
</tr>
</tbody>
</table>
EXAMPLES OF APPLICATION

Materials

Some of the investigated materials are described in Table 1, which also displays attenuation coefficients and absorption coefficients measured at a frequency of 5 MHz. It can be seen that the absorption coefficients are much lower than the corresponding attenuation coefficients which have been determined by conventional techniques. Here, this is due to the fact that the attenuation coefficients are dominated by scattering losses produced by the relatively large grain sizes of the specimens. However, it should be mentioned that in the case of very fine-grained steels the absorption losses might dominate the total attenuation especially at lower frequencies.

Elastic deformation

The absorption coefficient $\alpha_A$ in a bar of austenitic Alloy 800H (X10 NiCrAlTi 32 20) was measured as a function of elastic strain by applying stresses up to one third of the yield strength. As can be seen from Fig. 3, the absorption increases with increasing strain and goes back to the initial value if the stress is released. This behaviour may be explained by the theory of dislocation damping yielding for the absorption due to the interaction of the ultrasonic wave with the dislocations /5/

$$\alpha_A \sim \Lambda \cdot L^4 \cdot f^2$$

(3)

for frequencies far below the resonance frequency of the dislocations. Here, $f$ is the frequency, $L$ is the dislocation length, and $\Lambda$ is the dislocation density. The elastic deformation leads to a bowing-out of those dislocations that are suitably oriented to the applied load. According to equation (3), the resulting increase in the loop length should cause a higher absorption in qualitative agreement with the experimental results.

Plastic deformation

The dislocation density in a polycrystalline material can be strongly enhanced by plastic deformation. Fig. 4 shows the dependence of the ultrasonic absorption coefficient on the plastic strain in Alloy 800H for two different heat treatments. In the as-received condition the material was solution annealed (1130°C/30 min/water), i.e. most of the carbon was in solution. A distinct increase in absorption is observed with increasing plastic deformation leveling off at plastic deformations above 10%. If the material is aged 16h/800°C, precipitation of carbides takes place. During plastic deformation, the carbides act as sources for dislocation multiplication. Consequently, a higher dislocation density is expected during deformation for this material state compared to the as-received state. This is exactly what one observes by means of absorption measurements (Fig. 4) showing that the ultrasonic absorption increases much stronger for the aged material.
Fig. 3  Normalized ultrasonic absorption coefficient $\alpha_A / \alpha_A^0$ as a function of elastic strain $\varepsilon_{el}$ in Alloy 800H.

Fig. 4  Ultrasonic absorption as a function of plastic strain $\varepsilon_{pl}$ in Alloy 800H for different heat treatments. After the deformation the specimens were solution annealed.

After the deformation, the specimens were solution annealed allowing the microstructure to recover. This is clearly indicated by a drop of the absorption coefficients to nearly the initial values (Fig. 4). It must be emphasized that the changes in ultrasonic absorption measured here (and also in most cases described below) did not lead to significant changes in the total attenuation coefficients as obtained from the changes in the total attenuation coefficients as obtained from the evaluation of backwall-echo sequences. An example of the change of the reverberation signal due to plastic deformation is shown in Fig. 5c.
Dislocation mechanisms play an important role during high-temperature creep (see for example /11/). During early creep one usually observes the generation of dislocation networks forming subgrain structures. Ultrasonic absorption measurements were performed on specimens of Alloy 800H which are creep-tested at 800°C/31 MPa. In order to take non-destructive measurements, the tests which are still in progress were interrupted every 2000 hours /12/. Fig. 6 shows the measured ultrasonic absorption coefficient as a function of creep strain. The absorption coefficients are normalized to the absorption coefficients measured in a reference specimen. At strains of approximately one percent, $\alpha_A$.

Fig. 6 Ultrasonic absorption in Alloy 800H as a function of creep strain (tests still in progress)
increases by a factor of about two. Around strain values of 1%, steady state creep starts in the investigated material /13/. Here, TEM-investigations have shown that dislocation networks develop between the range of minimum creep rate and the end of steady state creep under the considered conditions /14/. Thus, we believe that the observed change in ultrasonic absorption is caused by the change in dislocation structure.

Influence of magnetic fields

In addition to the losses caused by dislocation damping, one observes in ferromagnetic materials losses due to the interaction of the ultrasonic wave with the magnetic structure. The ultrasonic wave induces changes of the magnetization by Bloch wall movements as well as by rotation processes. This leads to the generation of micro-eddy currents and hence to the absorption of ultrasonic energy. Theory /7/ yields for the frequency dependence of the ultrasonic absorption coefficient $\alpha_A^M$

$$\alpha_A^M \sim \frac{f^2}{f_0} \frac{1}{1+(f/f_0)^2}$$

Here, $f_0$ is the frequency at which the penetration depth of the micro-eddy currents becomes comparable to the mean domain size. For rotational processes, $f_0$ is by a factor of about 10 higher than in the case of wall movements because a different magnetic permeability enters into equation (4) /10/. In the magnetic saturated state all magnetic losses should vanish.

Fig. 7 shows the ultrasonic absorption measured in a ferritic steel (specimen 1) and a perlitic steel (specimen 4, see Table 1) as a function of magnetic field strength. $\alpha_A$ drops drastically at very low fields and then slightly decreases up to the highest fields applied. A completely different behaviour at low fields is observed for the perlitic steel (Fig. 7). Here, the absorption increases at low fields, reaches a maximum, and decreases at higher fields similar to the case of the ferritic specimen. The effect becomes more pronounced at higher frequencies. The findings described above were typical for both kind of steel types.

![Fig. 7 Ultrasonic absorption as a function of magnetic field. (Dashed line: ferritic steel, 0.05 wt% C, solid lines: perlitic steel, 0.65 wt% C) $H_t$- tangential magnetic field strength](image-url)
Without applied magnetic field the main contribution to the magnetic part of the ultrasonic absorption should be explained by the interaction of the ultrasonic wave with the Bloch walls. In the ferritic steel the Bloch walls are easily movable thus leading to a high absorption. In contrast, the perlitic structure consisting of small lamellas of ferrite and cementite seems to hinder the movements of the walls. Actually, there is no magnetic contribution to the absorption at zero-field as indicated by the measured absorption at "saturation". With applied field, the Bloch walls become movable. At the same time the Bloch wall density decreases. The combined action of both processes could explain the maximum in Fig. 7. The decrease of absorption measured at higher fields clearly indicates the presence of rotational processes because the Bloch wall density goes to zero in that range. This is confirmed by the fact that the observed decrease follows the decrease of the magnetostriction at higher fields. Examples of the measuring signals with and without magnetic field are shown in Fig. 5a,b.

Frequency dependence

The measured frequency dependence of the ultrasonic absorption between 2 MHz and 15 MHz is shown in Fig. 8 for different steels without magnetic field and with "saturation" field. In the latter case, the absorption should be mainly due to dislocation damping. Then, for the ferritic steel as well as for the perlitic steel and the austenitic steel (Alloy 800H) the frequency dependence seems to be quadratic in agreement with equation (3) but taking into account the measuring error of about ± 10% a linear dependence cannot be excluded. A linear frequency dependence is found for the ferritic-perlritic steel (specimen 2, see Table 1). The magnetic part of the absorption, i.e. the difference of \( \alpha_A \) measured without and with saturation field, depends linearly on frequency for both the ferritic and the ferritic-perlritic steel in the investigated frequency range. Concerning the perlitic steel (Fig. 8a) the absorption is higher at a field strength of 700 Oe than without field. This indicates that there is no complete saturation at 700 Oe as well as that there is no magnetic contribution to the ultrasonic absorption at zero field.

![Fig. 8 Ultrasonic absorption in different steels (see Table 1) as a function of frequency without and with magnetic saturation.](image)
a) solid lines: specimen 1, dashed lines: specimen 5
b) solid lines: specimen 2, dashed lines: specimen 4
CONCLUSIONS

The method presented here allows the direct measurement of ultrasonic absorption in polycrystalline materials which scatter ultrasound sufficiently. It can be used with a high sensitivity to study changes of the dislocation structure as well as the magnetic structure of materials, especially steels. This might be important for the nondestructive evaluation of materials properties. Additionally, the proposed method enables a separation of scattering losses and absorption losses when combined with conventional attenuation measurements.

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REFERENCES