INTERNAL FRICTION USE IN MONITORING THE DEGRADATION PROCESSES IN A MATERIAL

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Resume

During the measurement of the fatigue life of material, the specimen undergoes large number of stress cycles. While the observed number of cycles before failure is considered to be the most important result (often the only one), more knowledge can be obtained. Measuring energy consumption of specimen during the cycling, the changes of microstructure of material (especially dislocation structure and crack initialization) can be traced. The evolution of dynamic modulus can be watched on the same equipment. These information becomes very important, especially in the research of gigacycle fatigue strength and fatigue limit below which the continued loading does not lead to structural failure. In this article some remarks on microstructural changes in materials, which can be monitored by nondestructive measurement of internal friction are presented.

Available online: http://fstroj.uniza.sk/PDF/2011/25-2011.pdf

Article info

Article history:
Received 9 August 2011
Accepted 30 September 2011
Online 24 October 2011

Keywords:
Microstructure
Internal Friction
Fatigue
Dislocation-solute atom interaction
Heat treatment

ISSN 1335-0803

1. Introduction

The methods of internal friction (IF) measuremens are developed and widely known. These nondestructive testings bring us a wide spectrum of knowledge, but the excessive abstractness of obtained data hinders from larger interdisciplinary use of these methods. The IF spectrum often contains unique microscopic information that cannot be obtained by other methods. Due to the large variety of phenomena, materials and microscopic models, a correct interpretation of measured IF spectrum is often difficult [1,2].

For practical interdisciplinary use of IF techniques it may be not important to do an interpretation of spectrum. In the case of the continual measurements of the fatique life of material, for example, it is not possible to measure any spectrum. Time evolution of IF at constant experimental conditions (temperature, frequency and strain amplitude) is obtained instead.

The necessary knowledge about the relationship between the evolution of IF and evolution of microstructure must be found otherwise. This apriory relationship may be obtained by supporting measurement of IF spectra in advance. Once identified impact of microstuctural parameters on IF in the expected experimental conditions enables us to follow the evolution of microstructure during the experiment.

Four examples of above approach are presented here. First, the impact of dissolution of segregated intermetalic particles and material homogenisation on IF of magnesium alloy AZ 31 at elevated temperatures is introduced. Next, the impact of precipitation of cementite in low carbon feritic steel is shown. Then, the impact of dislocation structure changes on IF is handled.

And last the impact of micro-cracks on IF is presented.

2. Material and experimental method

All presented measurements were conducted in an ultrasonic test system with a nominal resonant frequency of 20.5 kHz, see Fig.1. This axi-symetric system consists of a piesoceramic ultrasonic transducer, horn, titanium rod and magnesium specimen. Vibration amplitude is measured indirectly using the amplitude of electric current flowing the transducer. Amplitude and frequency of loading voltage is controlled by computer. Temperature of specimen is controlled by oven, while the temperature of transducer must be constant (i.e. room temperature). Dimensions of all mechanical vibrating components are adjusted to vibrate in resonance. The acoustic length of equipment is integral number of half wavelengths λ/2. The acoustic length of specimen is exactly λ/2, with stress amplification shape to ensure maximal strain in the mid-length of sample.

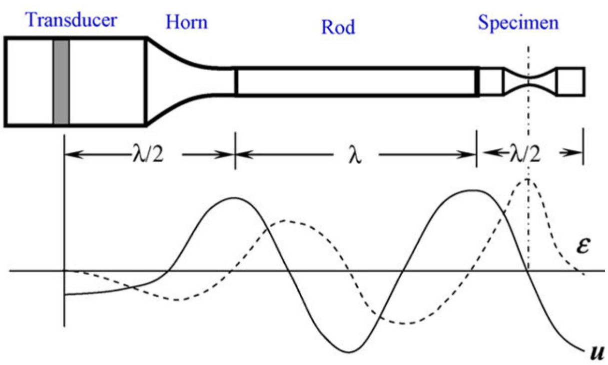


Fig. 1. Ultrasonic testing system[3]

A major advantage of the presented equipment is its multifunctionality. The same system can be used for both the measurement of the fatique life of material and the measurement of IF. What is more, the heat treatment of specimen can be done in the equipment during the measurement.

The method of resonant system quality Q determination was used for measurement of IF. By this method the half width of resonance peak in half height is measured. Even if the mass of equipment is hundred times greater than the mass of specimen, this method has a great sensitivity of the order 10^{-4}. The amplitude dependence of the IF of equipment must be negligible compared with the IF of sample.

The magnesium alloy AZ31 was produced by the squeeze-casting method at Brandenburgische Technische Universität, Cottbus, Germany. The chemical composition is in Table 1. As-cast structure was dendritic with different phase colonies in interdendritic regions. An impact of heat treatment on structure and IF is discussed later.

Table 1

Chemical composition (in wt. %) of AZ31 alloy

Al	Zn	Mn	Ca	Mg
3.62	1.36	0.29	0.18	Base

Low carbon high quality steel 12014 (marked by EN norm RFe 80) was used for presentation of impact of precipitation on IF. The chemical composition of the material is shown in Table. 2. The steel was supplied in the form of bars with gauge diameter 15 mm. From these bars the cylindrical specimens were machined for experiments. The original structure was polyhedral ferritic – pearlitic with pearlite content approximately 5 vol.%. The heat treatment of specimen was performed at the temperature T = 750 °C, holding at this temperature for 1 hour and consequently quenching into the water. The result of this heat treatment was the carbon dissolved in ferrite matrix at the maximum solubility about 0.02 wt%.

Table 2

Chemical composition (in wt. %) of 12014 steel

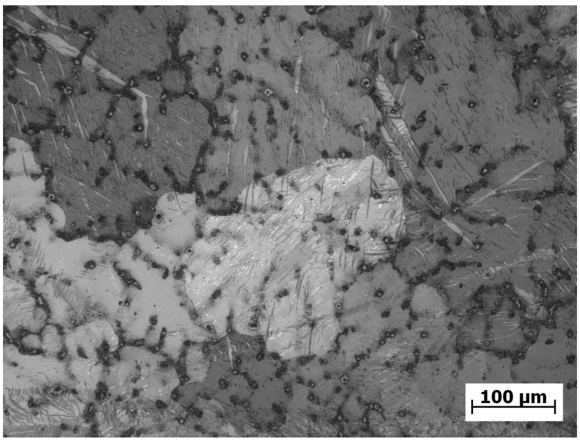
C	Mn	Si	P	Fe
<0.06	<0.45	<0.15	0.02	Base

3. Results

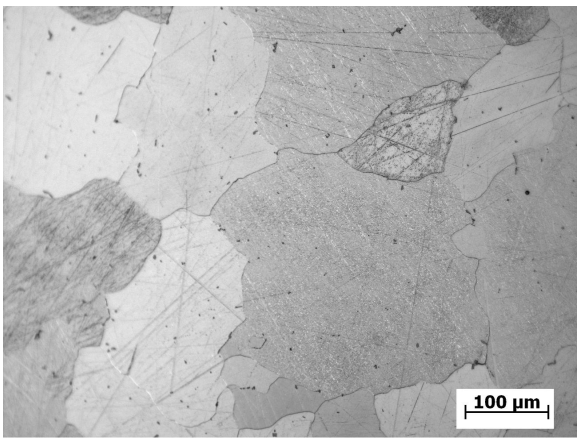
3.1. Impact of dissolution of intermetalic phases on IF of AZ31 at elevated temperatures

The IF spectrum of AZ31 was found to be composed of a low temperature background under 150°C which does not depend on temperature and of the IF peak at about 300°C which evolves into a rapidly rising background at higher temperatures. This spectrum is explained in terms of a solute-dislocation interaction model with a result that both the peak and background at higher temperatures could be interpreted by the high temperature dragging of substitutional atoms by dislocations [4].

Measurements [5] have shown, that IF above the peak temperature 300°C is very sensitive to changes in microstructure, especially to the changes of concentration of soluted atoms in magnesium matrix. The IF spectrum have been repeatedly measured three times on the same specimen (machined from the as-cast material without heat treatment, Fig. 2a.) in the 25°C – 450°C temperature range with heating rate 0.9°C/min. The original microstructure of material has changed from dendritic with segregated intermetalic particles to polyedral with homogeneous chemical composition (Fig 2b.) during the measurements.



a)



b)

Fig. 2. Microstructure of AZ31 specimen a) initial state, b) final state

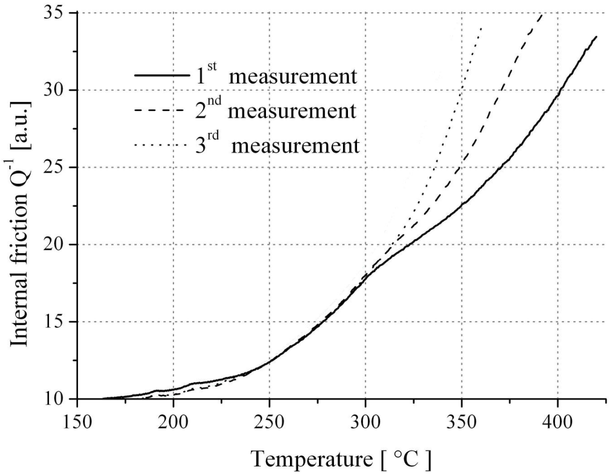


Fig. 3. Internal friction spectra of AZ31

The results are in Fig.3. The high temperature background is growing with growing concentration of soluted atoms while peak disappears. This was confirmed on second specimen with the same initial state. The measurement of IF was performed at constant temperature 400°C for 520 minutes. The IF was growing exponentially with second root of time, what is typical for diffusion processes. The obtained characteristic time was 64 minutes. Subsequent metallographic analysis has shown, that 400°C is not sufficient temperature to dissolve all intermetallic particles in 9 hours.

This phenomenon can be advantageously used to monitor the kinetics of structural changes of materials at elevated temperatures.

3.2. Impact of the precipitation of cementite in low carbon feritic steel

In body centre cubic (BCC) materials, interstitial solute atoms are found in octahedral sites, leading to local strain distortions. These distortions exhibit anelastic relaxation, due to a reorientation mechanism [2]. Snoek [6] first discovered this relaxation by measuring the internal friction of a Fe-C sample and he found that damping was proportional to the carbon concentration. The Snoek relaxation contributes to the IF spectra by the so-called Snoek peak which is of Debye type.

Reorientation mechanism requires only short-range diffusion, hence this process is thermally activated and peak temperature is related to the measuring frequency by Arhenius equation [2]. At 20 kHz the Snoek peak is located between temperatures 75 °C up to 350 °C with maximum at 207 °C, so this frequency is particularly suitable for monitoring the process of precipitation of cementite in oversoluted ferrite.

The kinematic of precipitation process running in steel 12014 was observed at the temperature 220 °C. Firstly two calibration measurements were done. The IF spectra of the specimen with oversaturated α-Fe with the carbon concentration 0.02 wt%. was measured immediately after heat treatment at 750 °C for 1 hour. Secondly, the spectrum was measured on the same specimen after the precipitation of cementite had finished at the temperature 350 °C. The relation between carbon concentration and IF was calculated by mutual subtracting of both measured spectra and some numerical post processing. Apart from the Snoek peak at the temperature 207 °C, the other one was found at 170 °C. Both of them are marked with arrows on Fig. 4.

For observing the precipitation of cementite, the same specimen was used as for calibration. Oversaturated state was refreshed by the heat treatment at 750°C for 1 hour again. The sample was heated to the required temperature 220°C by the rate about 5°C/min. Next, the IF evolution of material was recorded for 5 hours at constant temperature.

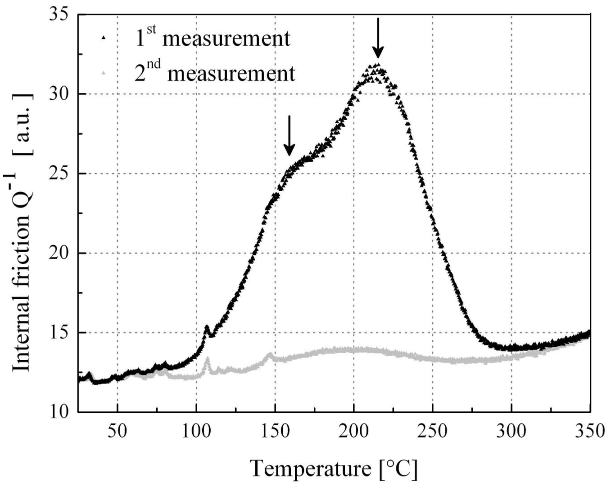


Fig. 4.Calibration measurements of IF on steel 12014

The IF was decreasing exponentially with second root of time, what was expected, because the precipitation of cementite is long-range diffusion process. By fitting the measured data, the relaxation time of precipitation τ = 2000 s was obtained. The exponentially increasing resonant frequency was also detected because of higher Young´s modulus of cementite [7].

3.3. Impact of the dislocation structure changes on IF

The changes in dislocation structure are the most natural results of cyclical stress. At low temperatures the anelasticity of dislocations comes from pining-depinig mechanism. The amplitude dependence of IF of the metal alloys is described by Granato-Lücke formula

$$Q^{-1} \approx Q_0^{-1} + \frac{\Delta}{\pi}\frac{\varepsilon_{cr}}{\varepsilon_0}\exp\left(-\frac{\varepsilon_{cr}}{\varepsilon_0}\right) \qquad (1)$$

where ε_{cr} is an characteristic strain amplitude which involves critical force on the obstacle at breakaway, the dislocation loop length between anchoring points, the segment length between obstacles and Burges vector and Δ is relaxation strength [2].

The impact of changes in dislocation structure on the IF was measured on magnesium alloy AZ31 at the temperature -15°C. The specimen was machined from the as-cast material and subsequently heat treated at 470°C for 10 hours, resulting in polyhedral structure of material (like Fig. 2b) with fully recovered dislocation structure. Five measurements of amplitude dependent internal friction (ADIF) were done, each of them was preceded by cycling at chosen strain amplitude ε_c for 10 minutes followed by 30 minutes pause to settle the microstructure.

Measured data have been fitted by formula (1) with results presented in Table 3.

Table 3

Results of Granato-Lücke fit (1) of ADIF spectra

ε_c (%)	Q_0^{-1} (a.u.)	ε_{cr} (%)	Δ / π (a.u.)	$\varepsilon_{cr} / \varepsilon_c$
0.17	12.5	0.80	60	4.7
0.19	12.6	0.84	60	4.4
0.22	12.4	0.99	85	4.5
0.24	12.3	1.08	89	4.5
0.33	12.5	1.42	93	4.3

The relationship between the cycling amplitude ε_c and characteristic strain amplitude ε_{cr} is evident from the last column of Table 3. The dislocation density (which is proportional to the relaxation strength Δ) is growing rapidly beyond the cycling amplitude 0.20 %. Resultant effect of these opposing effects is a decrease in the IF of material exposed to cyclic stresses.

In general, the dependence of IF on the number of cycles at constant cycling amplitude may exhibits four regions: damping increases at beginning, then steady state followed by rapid decrease of damping and finally rapid increase of damping caused by cracks nucleation [8].

3.4. Impact of the micro-cracks nucleation on IF

A simple rheological model taking into account the crack origin of damping was developed by Goken and Riehemann [9]. According to the model, one elementary crack is assumed to be represented by a frictional grip that is attached in series to a spring E_1 representing the loss of modulus due to opening of the crack. The grip is resistant to the stresses lower than critical stress σ_{c1} at which the crack opens.

In this model, which is valid for low frequencies, the amount of dissipated energy of one open crack during one cycle depends only on the value of its effective parameters such a

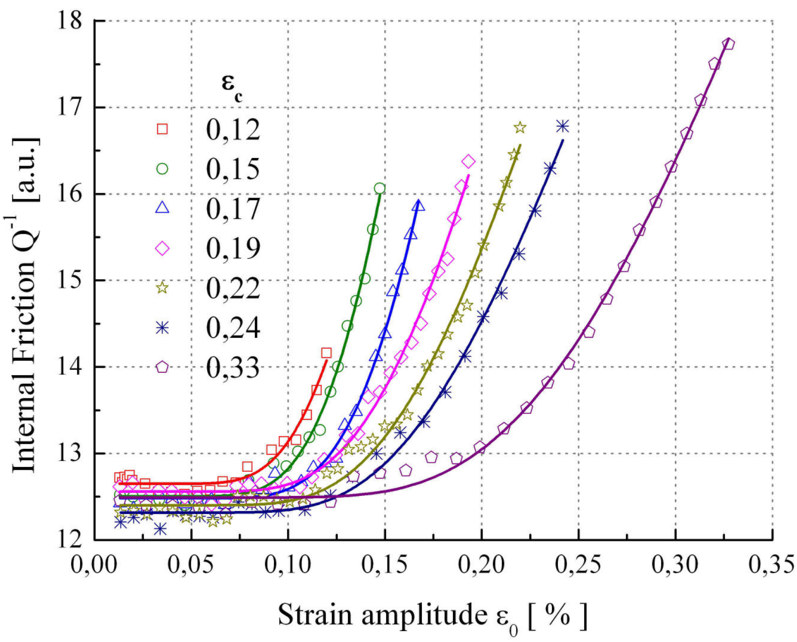


Fig. 5. Amplitude dependent IF measured on AZ31 at the temperature -15°C after cycling at strain amplitude ε_c Symbols represent measured data, lines comes from Granato-Lücke fit.
(full colour version available online)

critical stress of grip and spring constant but does not depend on the stress amplitude or the frequency. With growing size of the crack, the critical stress monotonously decreases.

At higher frequencies it is necessary to add a further contribution to the model which is derived from kinetic energy dissipated during the closing of crack. Kinetic energy is proportional to the second power of frequency and stress amplitude.

In summary, when the stress amplitude is lower than the σ_{c1}, the crack is closed and does not contribute to the IF. When the stress amplitude is higher than σ_{c1}, the contribution of crack to the IF consists of two parts:

$$Q^{-1} \cong \alpha / \sigma^2 + \beta f^2, \qquad (2)$$

the first one is described by Goken and Riehemann model [9] with IF reciprocal to second power of stress amplitude, the second one is proportional to the second power of frequency. The parameters α, β depend on crack size.

The IF in the 4th region is rising, as the nucleated micro-cracks are growing and more and more of them have critical amplitude smaller than the actual cycling amplitude.

4. Conclusions

The methods of internal friction (IF) measurements are non-destructive, well developed and bring us a wide spectrum of knowledge. Especially the measurement of fatigue life of material, use the same equipment as the measurement of IF.

By measuring the energy consumption and dynamic modulus changes, the microstructural changes can be traced. This knowledge may be very important. Four special cases of microstructural changes and their impact on IF are presented in this article. First two examples can be used for heat treatment preparation of specimens, while the last two cases are very useful for monitoring the structural changes of material during fatigue tests.

Each of these four examples presents a drift of IF which is due to different one mechanism. The dissolution of precipitates at high temperatures influences the IF due to solute-dislocations interaction. The Snoek effect originates in reorientation mechanism caused by short-range diffusion of interstitials atoms. The impact of dislocation structure on IF is described by Granato - Lücke model, changes of IF during cycling comes from dislocation-dislocation interaction. The evolution of microcracks in material can be monitored due to the existence of critical stress, at which the crack opens itself, and also because the maximal contribution of the crack to the amplitude dependent IF arises at the critical stress.

All these information become very important, for example in the research of gigacycle fatigue strength and fatigue limit.

Acknowledgements

This work has been supported by Scientific Grant Agency of Ministry of Education of Slovak republic, No. 1/0249/09.

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