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APPLICATIONS OF MECHANICAL SPECTROSCOPY TO INDUSTRIAL MATERIALS

ZASTOSOWANIA SPEKTROSKOPII MECHANICZNEJ DO MATERIAŁÓW PRZEMYSŁOWYCH

The paper is a review of original results, which were obtained by mechanical spectroscopy in the development of industrial materials, such as grey cast iron (damping capacity), aluminum alloys (recrystallization), nickel alloys (grain boundary embrittlement) and gold alloys (hardening mechanisms). Moreover it is shown that the study of grain boundary sliding at high temperature has led to the development of new grades of zirconia exhibiting a high toughness and a good resistance to creep. It is also recalled that mechanical spectroscopy has been a mandatory technique in the development of light metallic materials, which exhibit simultaneously good mechanical properties and a high damping capacity, and are consequently well suited to transport means.

Keywords: mechanical spectroscopy, internal friction, damping capacity, industrial materials

Artykuł prezentuje przegląd oryginalnych wyników, które zostały uzyskane za pomocą spektroskopii mechanicznej podczas rozwoju materiałów przemysłowych, takich jak żeliwo szare (zdolność tłumienia), stopów aluminium (rekrystalizacja), stopów niklu (kruchość granic ziaren), i stopów złota (mechanizmy umocnienia). Ponadto pokazano, że badania poślizgu granicy ziaren w wysokiej temperaturze umożliwiły rozwój nowych gatunków tlenku cyrkonu wykazujących wysoką wytrzymałość i dobrą odporność na pełzanie. Przypomniano także, że spektroskopia mechaniczna jest obowiązkową techniką stosowaną w rozwoju lekkich materiałów metalowych, które wykazują jednocześnie dobre właściwości mechaniczne oraz wysoką zdolność tłumienia, a tym samym są odpowiednie do zastosowania w środkach transportu.

1. Introduction

Mechanical spectroscopy has been advantageously used not only in fundamental research but also in research applied to materials, which have industrial applications [1]. However in this latter case, a question often arises from the project partners: what are the benefits to use such a complicated method instead of the classical techniques like, for instance, tensile tests, hardness tests, electrical resistivity measurements, electron microscopy, etc. The present paper aims at showing that mechanical spectroscopy has brought original results not only in the development of new materials, but also for improving the properties of "well-known" industrial materials, such as the damping capacity of grey cast iron, the recrystallized structure in aluminum alloys, the grain boundary properties in nickel alloys, the hardening mechanisms in gold alloys. More recently, the study of grain boundary sliding in fine grained ceramics has allowed the development of new grades exhibiting a high toughness and a good resistance to creep. Also mechanical spectroscopy has been a guide technique in the development of light metallic materials, which exhibit simultaneously good mechanical properties and a high damping capacity. As a matter of fact, a high damping capacity is good for decreasing the noise emitted by an engine, but also for improving the fatigue resistance of the vibrating components.

2. Experimental method

Even if industrial materials have a complex structure, their mechanical loss spectrum may be rather simple with one or two peaks superimposed on an exponential background. Using mechanical spectroscopy one has first to look for the characteristic mechanical loss spectrum of the material. Then, when it is possible to isolate the different phases, which compose this material, one has to measure the mechanical loss spectrum of each of them. For instance, the mechanical loss of grains in a polycrystalline material can be obtained by measuring a single crystal. By comparing the spectra of the components with the spectrum of the composite, it is possible to locate the source of the internal damping. The study of the dissipation mechanisms (point defects, dislocation or interface damping) is supported by the background accumulated during more then 40 years by the scientific community working in the field of mechanical spectroscopy or internal friction. Finally the interpretation of the spectrum at the atomic scale allows one to predict the material behavior in use (examples below).

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3. Results

3.1. Grey cast iron for machine tools

Grey cast iron is currently used in machine tool industry because of its high damping capacity. However, this material was characterized by the level of its ultimate tensile strength only (for instance, 250 MPa for the grade GG25) and materials scientists aimed at improving the tensile strength of grey cast iron, ignoring the damping capacity. In order to meet the needs of industry as it concerns the problems caused by undesirable vibrations, we investigated the damping mechanisms in grey cast iron [2, 3]. Systematic measurements of many grades showed that grey cast iron exhibits a characteristic mechanical loss spectrum, mainly composed of an abrupt increase of the internal friction between 180 and 250 K, associated with a modulus anomaly (GG25 in Fig. 1a). Comparison of this spectrum with the ones of the components (graphite and ferrite) allowed us to identify clearly the graphite phase as the source of the internal damping, the energy dissipation being due to dislocation motion within the graphite basal planes.

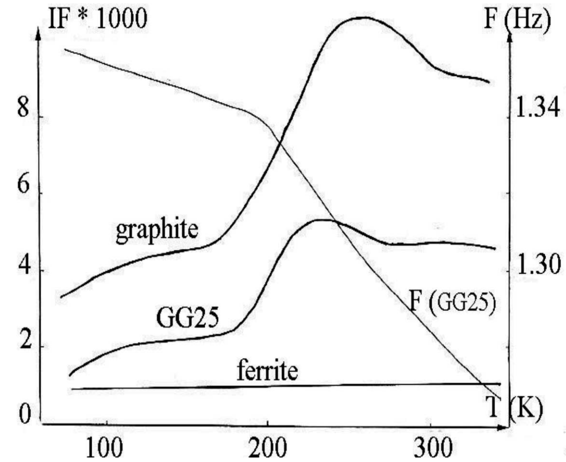


Fig. 1. Internal Friction (IF) spectra of GG25 grey cast iron, graphite and ferrite

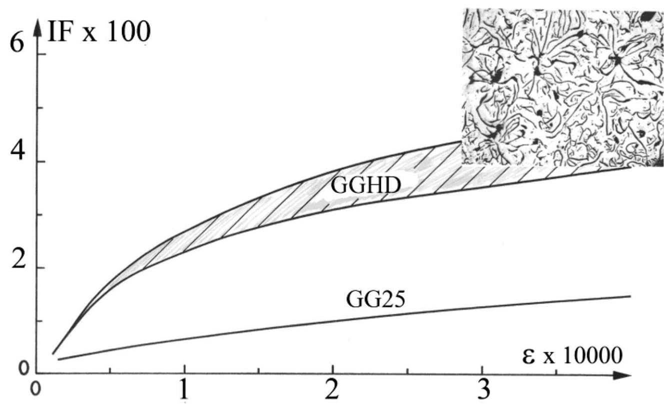


Fig. 2. Internal Friction (IF) as a function of the vibration amplitude ε in the GG25 grade and in a new high damping grade (GGHD) of grey cast iron

Considering the composite nature of grey cast iron with the graphite precipitates responsible for damping and the matrix (ferrite or pearlite) for the tensile strength, we were able to produce new grades of this material, which exhibited simultaneously good mechanical properties and a high damping capacity. As a matter of fact, the mechanical loss of a new industrial grade GGHD (High Damping Cast Iron) is compared to the one of GG25 in Fig. 2. The level of damping in GGHD for a vibration amplitude of 10^{-4} is well suited to the machine tool industry needs.

3.2. Light metallic materials for transport means

Transport means require lightweight materials that exhibit good mechanical properties such as high modulus, high mechanical strength, good creep or fatigue resistance, but also, in certain cases, a high damping capacity. One solution for optimizing the damping capacity and mechanical properties in a metallic material is the development of two phase composites, such as the Metal Matrix Composites (MMCs), in which each phase plays a specific role: damping or strengthening. Among the light metals, magnesium possess the highest damping capacity [4]. Adding ceramic reinforcements in a magnesium matrix allows one to improve the elastic modulus without lowering the damping capacity.

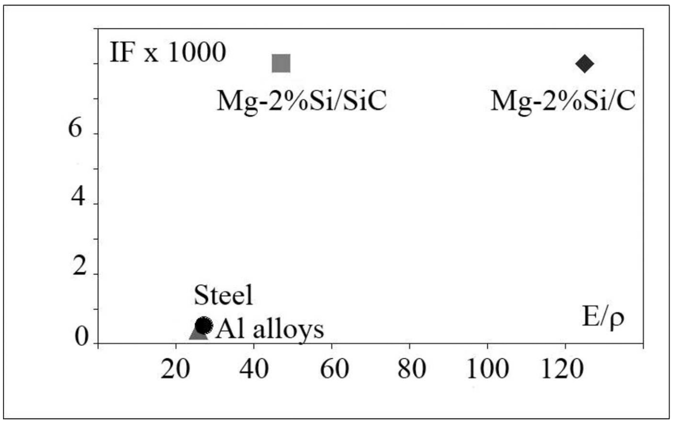


Fig. 3. Internal Friction (IF) as a function of the specific modulus of magnesium matrix composite in comparison with steel and aluminum alloys

Mg-2wt.%Si alloys reinforced with long unidirectional SiC or C fibers were processed by gas pressure infiltration and characterized by mechanical loss measurements [5, 6]. The Young's modulus, measured by means of a fee-free bar apparatus, was found to be 99 and 239 GPa in SiC/Mg-2wt.%Si and C/Mg-2wt.%Si, respectively. Remarkable are the specific mechanical properties (Fig. 3). The SiC/Mg-2wt.%Si and C/Mg-2wt.%Si composites have specific Young's modulus much higher than steel or aluminum alloys, with a damping capacity ten to hundred times higher.

3.3. Fine grained aluminum alloys for packaging industry

Al-Mn based alloys have been chosen for making thin packaging foils. Tightness of these foils requires a very fine grained structure of the alloy, which can be obtained by a good control of recrystallization. In this context mechanical spectroscopy has brought very interesting results because the mechanical loss due to dislocations is proportional to the product Λ l, where Λ and l are the dislocation density and loop length, respectively. The product Λ l gives then a measure of the total driving force for recrystallization: the stored elastic energy in the deformed region is proportional to dislocation density Λ, while the dragging force due to the solute atoms which pin the dislocations, is proportional to the inverse of the dislocation loop length (1/ l). As a consequence, the higher the mechanical loss, the easier is recrystallization.

In Fig. 4, two internal friction spectra are shown, which are associated with recrystallization in two Al-1%Mn specimens differing from one another by their thermo-mechanical treatments [7, 8]. A higher level of the internal friction (at ~420K) after precipitation in one grade leads to a lower recrystallization temperature (TR1 lower than TR2 by about 100 K) and consequently to a much finer grain structure.

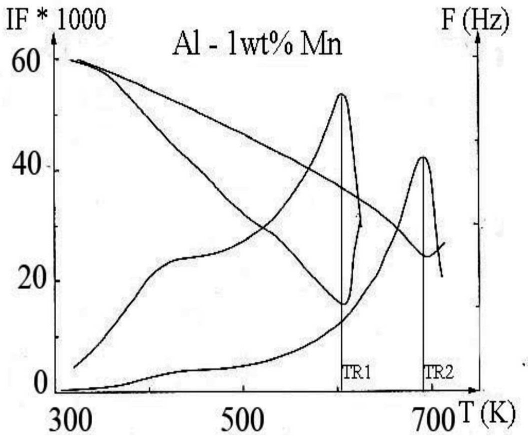


Fig. 4. Internal Friction (IF) and frequency (F) spectra of two Al-1wt.% Mn alloys with different thermo-mechanical treatments

3.4. Grain Boundary (GB) embrittlement in Ni – Cr alloys

The high temperature deformation and fracture behavior of polycrystalline materials are strongly affected by the presence of GBs and impurities. It is generally believed that GB sliding is an essential process, which causes stress concentration and, hence, cavity nucleation at obstacles along the GBs, such as triple points, ledges and second-phase particles. Accordingly the study of the structure and mobility of GBs is highly important for understanding the high temperature behavior of materials. The mechanical loss spectrum of polycrystalline Ni-20at.% Cr [11] is mainly composed of two peaks: P1 at ~950 K and P2 at ~1100 K for a 1 Hz frequency (Fig. 5a). In the spectrum of a single crystal of the same alloy only the P1 peak is observable (Fig. 5b). As a consequence, P1 peak originates within the grains and has been interpreted as a due to a Zener type relaxation. By comparing the spectra of mono- and poly- crystal, one can conclude that P2 peak is due to GBs. Moreover, it was observed that P2 peak exhibited a large hysteresis between the curves obtained upon heating and cooling (Fig. 5a). The hysteresis has been interpreted as due to the dissolution upon heating and precipitation upon cooling of GB precipitates, namely Cr_7C_3 carbides. After a thermal treatment in air (1 h at 700°C), the hysteresis disappeared; P2 peak being stable in height as well upon heating as upon cooling. This has been interpreted as due to the dissolution of the carbides by oxygen penetrating along the GBs giving rise to extensive GB sliding followed by microcracks formation at ledges and triple points [12]. Here the stabilization by oxygen of the GB peak in the mechanical loss spectrum has been correlated with the high temperature embrittlement of Ni-Cr alloys.

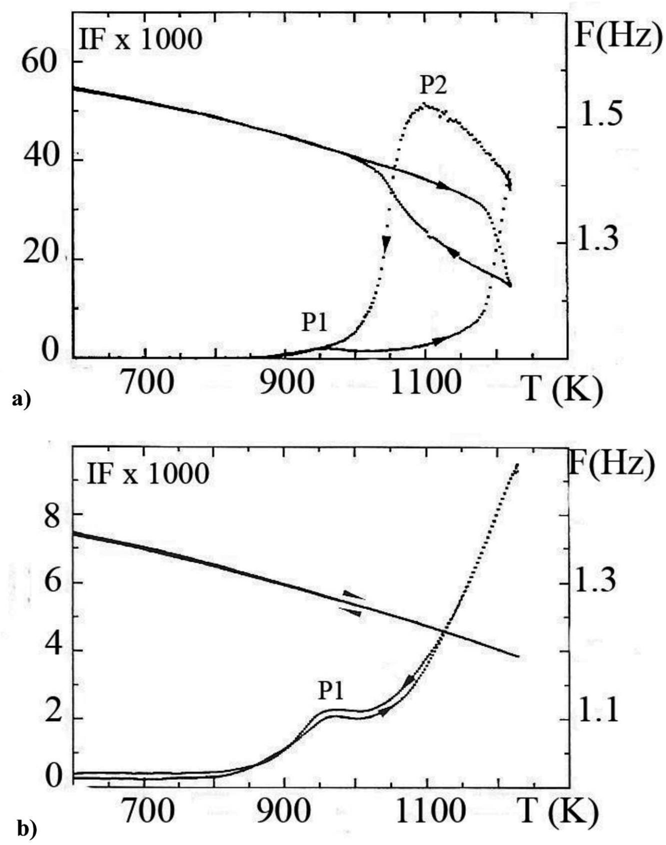


Fig. 5. Internal Friction (IF) spectrum of a) poly-crystalline and b) single-crystalline Ni-20at.% Cr

3.5. Hardening mechanisms in yellow gold alloys for watch industry

In order to improve the mechanical properties of industrially used 18-carat gold alloys, it is important to understand the hardening mechanisms, which are known to occur in these materials, such as atomic ordering and precipitation hardening.

In the medium temperature range, the spectrum of the yellow gold alloys presents a peak corresponding to a Zener relaxation. If the copper content in such an alloy is high enough, atomic ordering occurs, at temperatures below (roughly) 600 K. In the ordered structure, the Zener relaxation is no longer possible and, consequently, the peak breaks down [9, 10] (Fig. 6). The peak behavior provides a precise value of the order-disorder transition temperature (a phase diagram was drawn) as well as useful data on the transformation kinetics. Ordering with formation of a phase of tetragonal symmetry is responsible for the hardening of the yellow gold alloys.

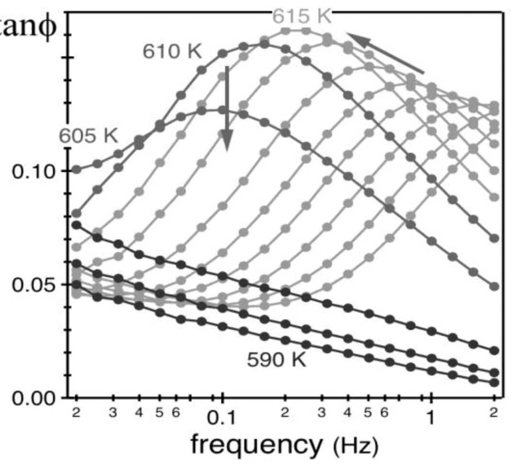


Fig. 6. The Zener peak breaks down in yellow gold alloys, when ordering takes place [10]

3.6. Hard metals for cutting tools

Ceramic-metal composites, like for instance WC-Co cemented carbides, are good examples of materials, which exhibit good creep resistance and good toughness. In Fig. 7a are reported the mechanical loss spectra of WC-11wt.% Co and of the same material after extraction of the Co binder phase by chemical etching [13]. A mechanical loss peak is observed in WC-11wt.% Co superimposed on an exponential high temperature background (curve 1). These two components of the spectrum have disappeared with the dissolution of Co by chemical etching (curve 2), and the conclusion is that the dissipation mechanisms take place in the cobalt phase. An exponential increase in the mechanical loss at high temperature means a bad creep resistance of the material, because the anelastic or microplastic strain has no limit. On the other hand, damping mechanisms are positive factors for improving toughness in hard and brittle materials. As a mechanical loss peak is associated with a restricted motion of structural defects, it cannot be associated with the onset of creep. It accounts for the ability of the material to dissipate locally a part of the vibration energy and hence to improve toughness by crack propagation blunting. As a matter of fact, the mechanical loss peak in WC-Co (Fig. 7a) appears in the same temperature range where an increase in toughness was observed [13]. In order to improve the mechanical resistance of WC-Co, alloying elements were added in the Co binder phase. The new grade exhibits a lower exponential high temperature background (Fig. 7b) and consequently a higher yield stress was observed at high temperature [14].

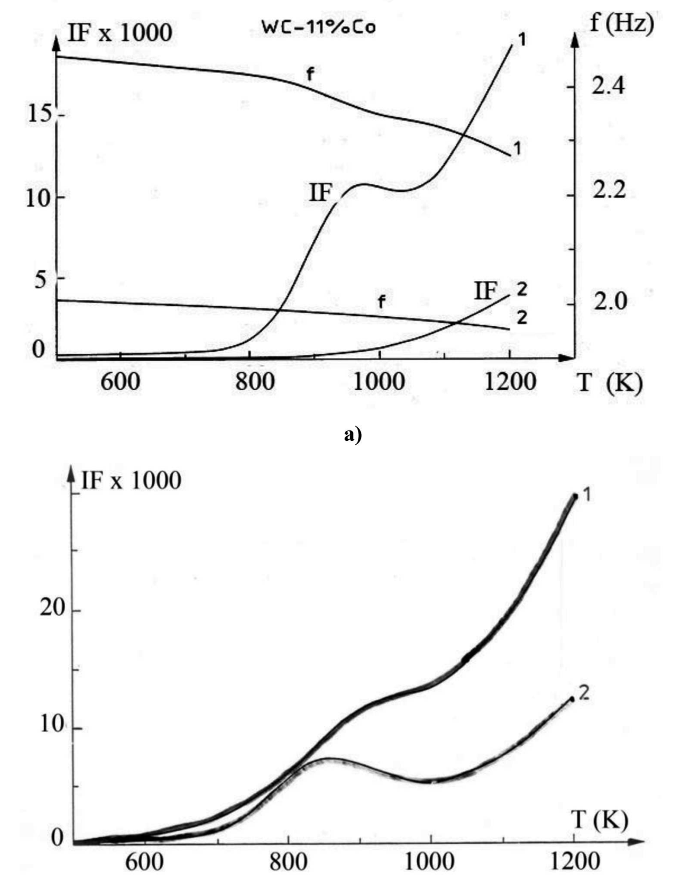
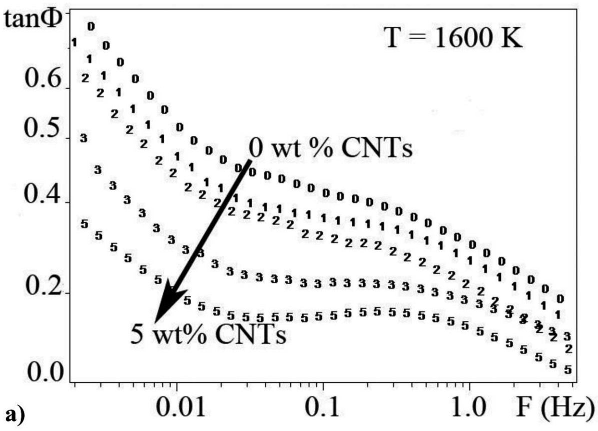


Fig. 7. a) Internal Friction (IF) and vibration frequency (f) of WC-11wt.%Co, as sintered (curves 1) and after removal of the Co binder phase by chemical etching (curves 2) [13]; b) Mechanical loss of WC-11wt.%Co (curve 1) and of WC-11wt.%Co + Cr, Ni (curve 2)

3.7. Fine grained ceramics with good toughness and good creep resistance

High temperature plastic deformation of fine-grained ceramics has been interpreted as mainly due to GB sliding. GB sliding, which is responsible for high temperature plasticity in fine grained ceramics, gives rise to a peak or an exponential increase in the high temperature or low frequency mechanical loss spectrum (Fig. 8a).

Fine grained zirconia was reinforced with carbon nanotubes (CNTs) in order to improve the restoring force acting in the GB sliding mechanism and consequently to improve the creep resistance [15]. The high temperature mechanical loss is remarkably reduced by CNT additions (Fig. 8a [16]). Compressive creep tests were also performed on parallelepiped samples under a stress of 8 MPa at 1600 K. Lower is the mechanical loss (Fig. 8a), lower is the high temperature (1600 K) creep strain (Fig. 8b). Such a behavior is interpreted as due to the pinning effect of the GBs by the CNTs. From the viewpoint of mechanical spectroscopy the brittle-to-ductile transition, which appears at high temperature in ceramics, can be decomposed into two stages: a brittle-to-"tough" transition associated with the mechanical loss peak and a "tough"-to-ductile transition associated with the exponential background. Structural ceramics have to be tough and creep resistant. In other words they have to exhibit a mechanical loss peak with a low level of the high temperature background. Composites made of fine grained 3Y-TZP zirconia reinforced with CNTs are good examples of such wanted ceramics.



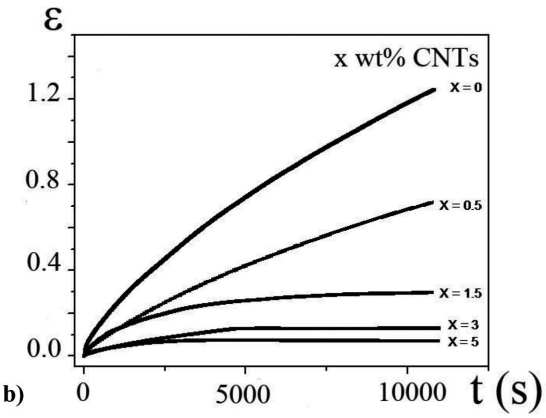


Fig. 8. a) Mechanical loss (tanΦ) as a function of frequency and b) creep strain (ε) as a function of time at 1600 K for 3Y-TZP zirconia reinforced with different amounts of CNTs

4. Conclusions

There are many technological problems, in which mechanical spectroscopy is a unique technique for finding the solution. It is our mission to interpret the mechanical loss spectra in order to demonstrate the advantages of the technique to our project partners, who are working with other more classical methods. The development of mechanical spectroscopy is not only related to the fundamental study of pure metals, but to the use of this technique to develop new materials and/or to improve the properties of existing industrial materials.

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