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## MECHANICAL SPECTROSCOPY OF BEARING STEEL

## SPEKTROSKOPIA MECHANICZNA STALI ŁOŻYSKOWEJ

This study presents mechanical spectroscopy of bearing steel subjected to different heat treatments. A non-thermally activated maximum, P1, was found at 130°C, in quenched martensitic samples, which were austenitized at 1050°C and 860°C, and presented twin martensite microstructures. It is suggested that the mechanism of the P1 maximum, observed on the low-temperature side of Snoek-Köster peak, is related to the change of defect configurations in twinned martensite assisted with high mobility of the solute carbon atoms under an external harmonic stress field applied during mechanical loss measurements.

*Keywords*: Bearing steel, twin martensite, carbon atoms, mechanical spectroscopy, internal friction, Snoek-Köster peak

W pracy przedstawiono wyniki badań spektroskopii mechanicznej stali łożyskowej poddanej różnej obróbce cieplnej. W hartowanych martenzytycznych próbkach, które były austenityzowane w 1050°C i 860°C, i posiadały bliźniaczą mikrostrukturę martenzytu, występuje nietermicznie aktywowane maksimum P1 przy 130°C. Sugeruje się, że mechanizm maksimum P1, które występuje na niskotemperaturowym zboczu piku Snoek-Köstera, związane jest ze zmianą konfiguracji defektów w bliźniaczym martenzycie, przy wysokiej ruchliwości atomów węgla. Maksimum P1 ujawnia się pod wpływem działania harmonicznie zmiennego zewnętrznego pola naprężeń przyłożonego do próbki w trakcie badań metodą spektroskopii mechanicznej.

## 1. Introduction

Quenched martensite is a supersaturated solid solution of interstitial carbon atoms inherited from the parent austenite [1]. Generally, the obtained martensitic material is hard and brittle. Thus, tempering is often required to tailor the toughness and hardness of the steel. It has been found [2-6] that the evolution of microstructures in high carbon steels with increasing the tempering temperature may involve segregation of interstitials and clustering, precipitation of transition carbides, decomposition of retained austenite, and transformation of the transition carbides into cementite.

Mechanical spectroscopy is considered as a powerful technique to obtain critical information concerning the dislocation-carbon interactions in steel from dislocation-based relaxation phenomena [7-24]. For example, mobile dislocation segments dragging along with them the Cottrell cloud of long-range mobile foreign interstitial atoms can be revealed by Snoek-Köster (SK) peak observed in deformed iron-based alloys [11-20]. The interactions between dislocations and short-range mobile carbon atoms can be assessed by the dislocation-enhanced Snoek peak in iron-based alloys [11,16,19-24]. It is important to note that the dislocation-enhanced Snoek peak is observed in ultra-high purity and technical purity Fe-C alloys containing very low concentration of carbon atoms [16,20,21,23]. Thus, not surprisingly the dislocation-enhanced Snoek peak is also observed in a commercial bake-hardening steel [24]. The Snoek-Köster and dislocation-enhanced Snoek peak are used for evaluating the amount of foreign interstitial atoms and the dislocation density. It is important to emphasize that Snoek-Köster peak (or equivalently, the cold-work peak) [11-16] occurs both in deformed ferrite [11-20] and in martensite [11,25-39].

It is well known that point defects in the octahedral sites of the body-centered cubic lattice (bcc) give rise to the well-known Snoek relaxation [7-9,11,40-48], which shows the characteristic anisotropic behavior [47,48]. However, the Snoek peak [49] and the dislocation-enhanced Snoek peak are absent in martenisite due to the tetragonality of the martensite. Instead, the carbon content in martensite may be assessed by the internal friction background [32]. It is recalled that carbon Snoek-Köster peak is frequently observed in ferrous martensite [25-39] in Fe-C alloys and numerous steels.

The evolution of mechanical loss spectra associated with the tempering of martensitic steels is complex. A few internal friction peaks were reported in high carbon tool steel (1.23

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wt.%C) and in medium carbon high alloyed steel [31-39]. A non-thermally-activated internal friction P1 peak at 130°C was first reported by Kê [25]. Lu [39] suggested that this peak stems from: (1) depletion of interstitial carbon due to the formation of transitional carbides or carbon segregations and abrupt decrease of the internal friction background [25,34-39]; (2) hysteretic depinning of dislocations or twin boundaries [39]. But nevertheless the physical nature of the P1 phenomenon (referred to as the P1 maximum) is still unclear due to the large difference in alloy compositions and in the substructures of the quenched martensitic phases reported in the literature [11,25-39].

It is interesting to recall that internal friction peaks observed in quenched, cold-worked and tempered Fe-Ni-C and Fe-C martensites were carefully reviewed by Klems et al. [37]. It should be emphasized that the internal friction peak at around 130°C in Fe-C alloy (0.8Cwt.%) was originally reported by Kê and Ma [31] in partially twinned, relatively untempered Fe-C martensites. Klems reported a relaxation peak at 160°C ($f_o\sim$1Hz) in Fe-Ni-C martensite, which was brought about by the stress-induced motion of twin boundaries containing mobile carbon atoms [37]. Klems concluded that the relaxation mechanism of Snoek-Köster peak (cold-worked peak) in deformed ferrite is the same as in ferrous martensite in steel [37].

In steels with carbon ranging from 0.6 to 1.0 wt. %, lath (with dislocations) and plate (with twins) martensite can be formed. The fraction of the plate martensite increases with increasing the carbon content [1-3]. The present work investigates mechanical loss spectra of a high-carbon (1.05C wt. %) bearing steel, which was austenized at different temperatures in order to adjust the carbon content and the substructures of the quenched martensite.

## 2. Experimental procedure

The material used in this study is a GCr15 bearing steel with the following chemical composition Fe-1.05C-0.27Si-1.46Cr-0.33Mn-0.002P (wt.%), to which a spheroidization annealing treatment has been applied. The specimens were quenched into oil (50°C, kept in oil for 5 min) after austenitizing for 10 minutes at 1050°C, 860°C and 820°C, respectively. Following this, selected quenched samples were subjected to tempering at 150°C for 2h and cryogenically treated in liquid nitrogen for 2h to induce further transformation of austenite into martensite. It is noted that the samples were stored at room temperature (RT) for less than 10h before internal friction measurements were performed.

Low frequency internal friction measurements were performed in an inverted torsion pendulum in the resonant mode. The pendulum operated at around 2Hz with the heating rate of 1°C/min from RT to 500°C. The internal friction, $Q^{-1}$, was calculated from the logarithmic decrement δ ($Q^{-1}=\delta/\pi$), estimated from the classic free decaying method [7,50-55]. The maximum strain amplitude was $2\times10^{-5}$. The specimen dimensions were $1\times3\times60$ mm³. An external magnetic field was not applied to suppress magnetomechanical damping.

Internal friction measurements at higher resonant frequency, $f_o$, ($f_o \approx$ 13Hz) were performed in a resonant instrument (Nihon Techno Plus Co., Ltd.) allowing the simultaneous measurement of the internal friction, $Q^{-1}$, and the resonant frequency, $f_o$. The internal friction was estimated from the classic free decaying method [7,50-55]. An electronic discriminator was used to record the number of oscillations within the range of the maximum strain amplitude to the half value. Young's modulus was determined from the resonant frequency, $f_o$, ($E\sim f_o^2$) using a commercial frequency meter. Mechanical loss measurements were performed in high vacuum in the temperature range from RT to 500°C at the heating rate of 1°C/min. Free decaying oscillations, at around 13Hz, were performed in bending. An external magnetic field was not applied to suppress magnetomechanical damping.

The free-decay oscillations are affected by the zero-point drift (ZPD) and offset [16,54-55]. It is tacitly assumed in this work that biased oscillations did not strongly affect the estimated internal friction values (the ZPD effect usually affects the shape of measured internal friction peaks and background level).

The light microscopy study was performed on polished samples after etching with 3% nital. For transmission electron microscopy (TEM) studies, similar samples were mechanically thinned up to 50–100 μm in thickness, and 3 mm discs were punched from the thinned plates and subjected to twin-jet electropolishing using an electrolyte containing 15% perchloric acid and 85% ethanol at ambient temperature and 40V potential (direct current). The microstructure of the specimens was studied in the JEM-2100F transmission electron microscope.

## 3. Results and discussion

Figure 1 shows internal friction spectra of the GCr15 bearing steel quenched samples, which were austenized at 820°C, 860°C, and 1050°C. The Snoek-Köster peak (denoted here as the P2 peak in martensite, Figs. 1,2) is observed at about 250°C in all the three samples. The P1 maximum is observed at around 130°C, in the 1050Q and 860Q samples only. It is noteworthy that all the samples have been freshly quenched. Consequently the carbon atoms and dislocations should be abundant in the martensite as clearly confirmed by the presence of Snoek-Köster peaks. Nevertheless, the P1 maximum is not observed in the 820Q sample, which is an interesting issue.
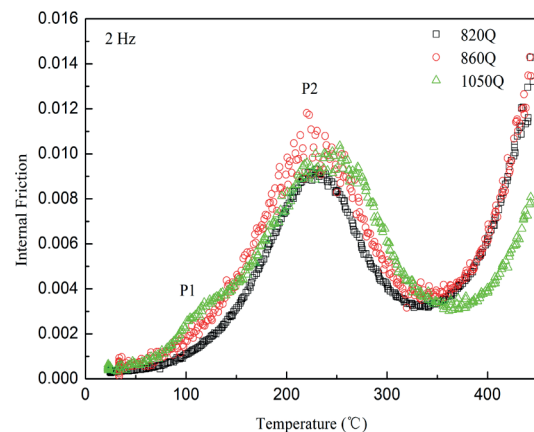


Fig. 1. Internal friction spectra measured at around 2Hz during the heating of freshly quenched samples after austenization at 820°C (820Q), 860°C (860Q), and 1050°C (1050Q)
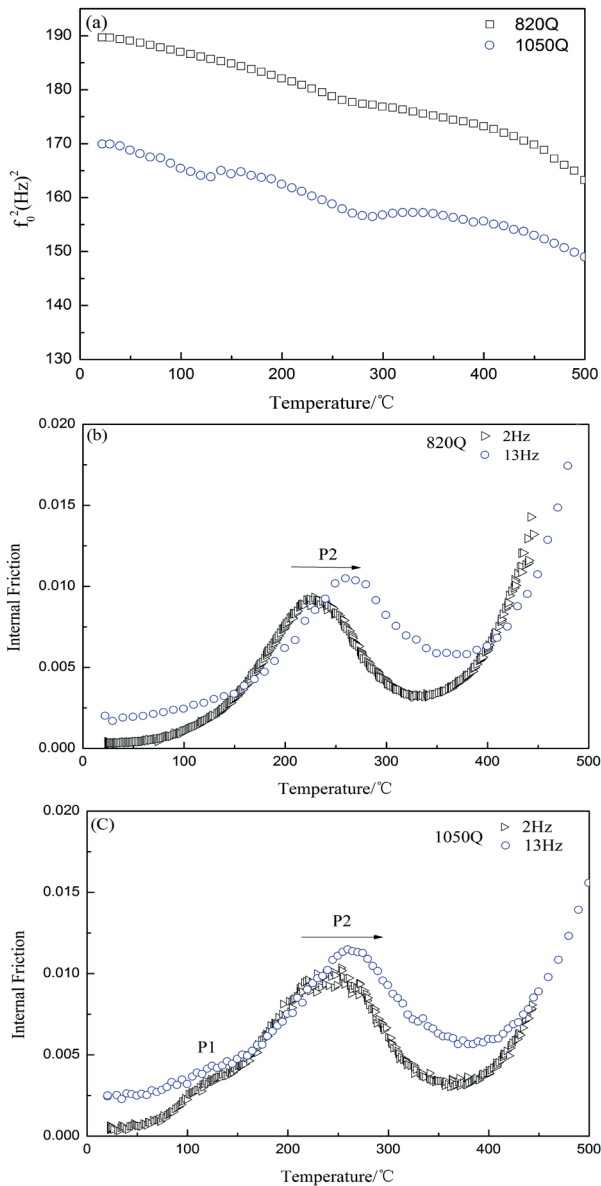
operating in bending, at around 13Hz, as compared to the inverted torsion pendulum operating at 2Hz. It is possible that the difference in background levels stems from different values of the maximum strain amplitudes used in two different instruments or from different algorithms used to compute the logarithmic decrement based on the counting of the number of free decaying oscillations [50,51,54,55].



Fig. 2. (a) Temperature variation of Young's modulus ($E \sim f_o^2$; where $f_o$ is the resonant frequency, $f_o \approx 13Hz$); (b) internal friction in 820Q, and (c) 1050Q samples

In order to find the characteristics of the P1 maximum mechanical loss measurements were also performed at higher frequency in the samples 820Q and 1050Q. As shown in Fig. 2a, a different slope of the elastic modulus is observed in the 1050Q sample around the P1 maximum at 13Hz. No such modulus variation is found in the 820Q sample. The Snoek-Köster (P2) peak exhibits typical relaxation-type behavior, i.e., the peak shifts towards higher temperature with an increase of the resonant frequency. By contrast, the P1 maximum is frequency-independent (Fig. 2c) indicating that the associated mechanism is not thermally activated as reported by Tkalcec [32]. It is noted that the P1 maximum is not observed in the 820Q sample (Fig. 2b.)

The internal friction background at room temperature is different in samples measured in two instruments used in this study. The background level is higher in the instrument
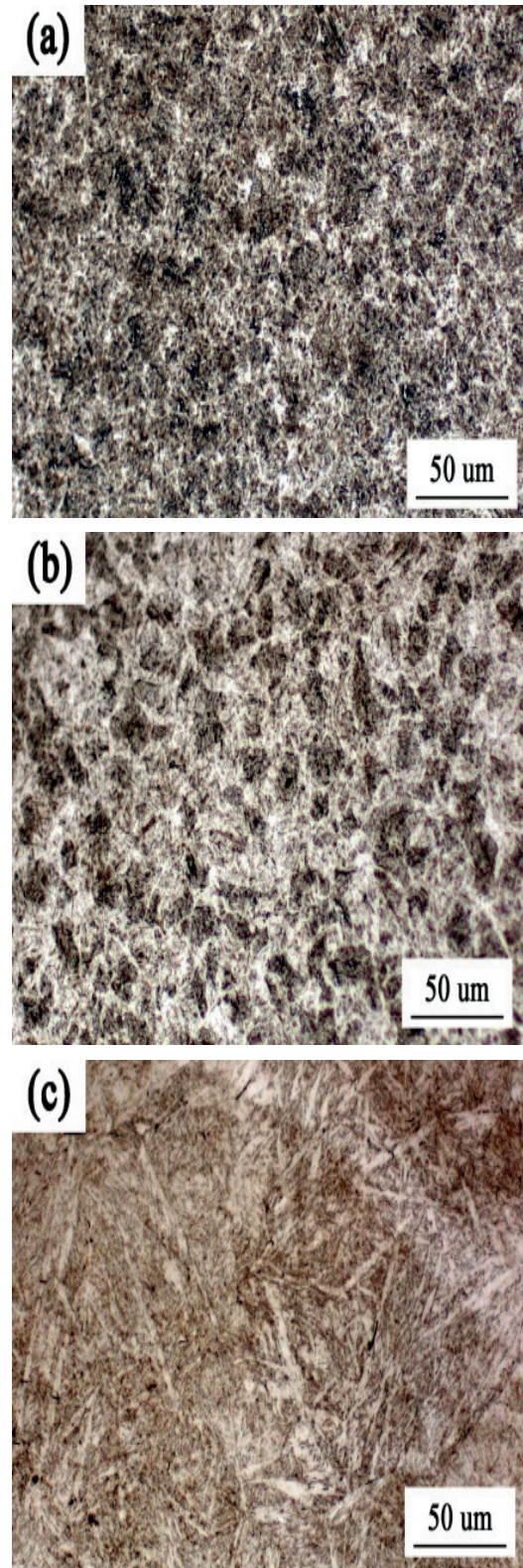


Fig. 3. The microstructure of freshly quenched samples (a) 820Q, (b) 860Q, (c) 1050Q

The microstructures of the 820Q, 860Q and 1050Q samples are shown in Fig. 3. One can observe that the fraction of martensite plates increases with increasing the austenitization temperature. The substructure of martensite mainly depends on the carbon content and the quenching temperature. Given that the phase transformation is accompanied by a volume change, plastic deformation can be accommodated by either twinning or dislocation mechanism [38]. It was reported that with increasing carbon content the fraction of martensite plates increases, and is accompanied by the formation of twin related variants in the martensitic phase [56,57]. In fact, a twinned microstructure is observed in the plate martensite of the 1050Q sample (Fig. 4, the habit plane is $(\bar{1}2\bar{1})$ ).
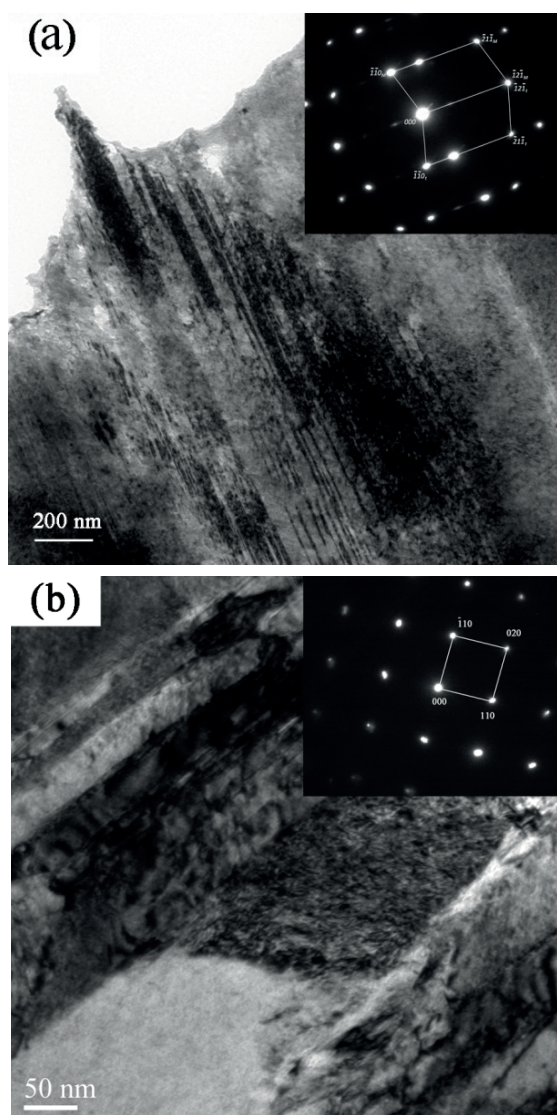


Fig. 4. TEM micrographs of (a) twining in plate martensite in the 1050Q sample, (b) dislocation structure in lath martensite in the 820Q sample

TEM micrographs of the 1050Q and 820Q samples are shown in Fig. 4. As expected, the typical microstructure of {112} twinning is abundant in the martensite observed in the 1050Q samples, while no precipitation can be identified from the diffraction pattern. The lath martensite is found in the 820Q sample containing a high density of tangled dislocations (Fig. 4b). Thus, it is presumed that the formation of ε carbides was excluded during the internal friction measurements performed between room temperature and 150ºC because of the relatively high heating rate.

In the internal friction spectra the P1 maximum temperature is close to the temperature at which the transition carbide forms. Carbon atoms behave as dislocation pinning points and the decrease of their density can lead to an increase of the free length of the dislocation loops. A possible explanation to the P1 maximum might be the reduction of the internal friction background that 'produces' an apparent P1 maximum. The non-thermally activated character may be attributed to the long-range interactions of edge dislocations with the elastic strain field induced by solute carbon atoms. Such a hypothesis seems reasonable when considering the P1 maximum of the 1050Q and 860Q samples in this study, but it is difficult to explain the absence of this maximum in the 820Q sample.

Another important fact is that the P1 maximum is related to the twinned microstructure. Thus, it is also possible to interpret the P1 maximum as due to the interaction between the carbon atoms and the twin boundaries in martensite. Let us assume that the mechanism is similar to the twin boundary-H interaction in Ti-Ni martensite [58]. Thus, a relaxation-type peak is expected to occur, which differs from the present experimental findings by the non-relaxation character of the P1 maximum. Therefore, another mechanism for the origin of the P1 maximum should take into consideration both the solute carbon and the twinned microstructure.

It is well known that mechanical twins can relax elastic energy stored in the matrix, concentrate applied stresses at their tips and transmit the stress over twin interfaces and into their interior [59]. Another possible manner of twin is to form different defect configurations (e.g. short edge dislocation segments) in various regions of a plate, for example, an inter-variant boundary with no long-range distortion field [60]. Based on the above facts and experimental evidence, an explanation for the P1 maximum could be anticipated. Firstly, we agree with the assumption that the internal friction background above room temperature stems from the long-range interaction of edge dislocations with solute carbon atoms [32]. Moreover, edge dislocations have possibly proliferated in the twin boundaries during plastic deformation. The P1 maximum could be then caused by the energy dissipation from the change of defect configurations in twin assisted by the presence of highly concentrated carbon atoms under an external harmonic elastic deformation giving rise to the fine modulus variation observed in Fig. 2a.

Cryogenic treatment in liquid nitrogen temperature after quenching (QC) is accompanied by plastic deformation of virgin martensite, which results in the capture of immobile carbon atoms [61,62]. The internal friction background in the 1050QC is higher than that in the 1050Q (Fig. 5). This effect may be due to the segregation of carbon atoms near the edge dislocations and also to the increase in the edge dislocations density in the twin boundaries of martensite. In martensite, where carbon and twin density are low, the P1 maximum is not observed. The P1 maximum may be then related to the change of defect configurations in high solute carbon martensite under an external harmonic elastic deformation. Further study is required to validate and advance this hypothesis.

Figure 5 shows a broad Snoek-Köster peak observed after quenching and cryogenic treatment. The width of Snoek-Köster peaks can be explained in terms of a lognormal distribution of relaxation times caused by the distribution of dislocation lengths [16]. The SK peaks observed after quenching and cryogenic treatment are broader than a single Debye peak. The SK peak in 1050QC sample is broader as compared to 1050Q sample. This suggests wider distribution in lengths of dislocation segments induced by cryogenic treatment. Further experimental and quantitative numerical analysis of internal friction peaks in martensite is required for the better understanding of the behavior of this peak in lath and plate martensites.
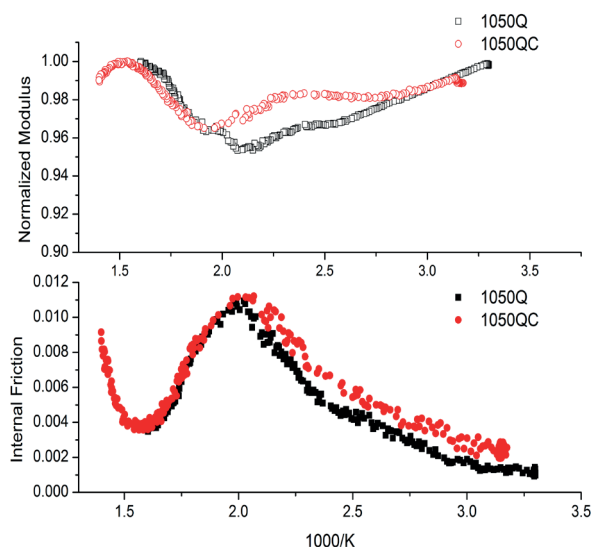


Fig. 5. Normalized modulus and internal friction spectra of 1050Q and 1050QC samples (the resonant frequency, $f_o \approx 2Hz$)

## 4. Conclusions

Mechanical loss spectra of the GCr15 bearing steel after different heat treatments were investigated. The mechanism of the P1 internal friction maximum, observed at 130ºC, is not yet clearly understood. It is proposed that the P1 maximum is brought about by the change of defect configurations in the twins in martensite assisted with the motion of high solute carbon under an external harmonic stress field applied during mechanical loss measurements. The presence of Snoek-Köster peak in bearing steel after quenching from 820°C, 860°C, 1050°C, and after additional cryogenic treatment confirms the interaction of mobile dislocation segments with carbon atoms in martensite.

### Acknowledgements

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