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Mechanical Spectroscopy of Rolling Oil Thin Films on Cold-Rolled Steel Sheets

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Abstract Fine traces of lubricating oil can be discovered in the form of fine debris left on the surface of cold-rolled steel sheets. High resolution mechanical spectroscopy is a sensitive technique for detecting such extremely fine oil traces. It is shown that a characteristic mechanical loss spectrum occurs in the low temperature range (from 180K up to 280K) only in the two following cases, namely (*i*) if traces of the rolling oil are left on the surface of sheets and (*ii*) if the clean sheets are covered with a thin film of the rolling oil (or any other natural or mineral oil). It is clearly demonstrated that similar mechanical spectra induced by the presence of oil can be observed in the sub-resonant mechanical spectroscopy if the oil film is deposited on a very soft cellulose neutral substrate. Therefore the behaviour of the oil alone can be obtained with a high resolution. It will be demonstrated that it is possible to assign the mechanical loss phenomena to the steel sheet (like the Snoek-Koster effect above 500K) and oil separately. In particular, it is shown that the low temperature relaxation phenomena do not originate from a specific interaction of the steel surface with oil, as frequently reported in the literature.

1. Introduction

High resolution mechanical spectroscopy (HRMS) is a very sensitive tool to study the dynamics of atomic or molecular motions in condensed matter. In the field of physical metallurgy, HRMS has been widely used to investigate relaxation processes related for example to dislocations (*e.g.* the Bordoni relaxation peak), point defects and point defects-dislocations interaction (*e.g.* the Snoek or Snoek-Köster relaxation process exhibited by ferritic steels, respectiveley).

This paper focuses on surface effects induced by the presence of lubricating oil remaining after cold-rolling followed by cleaning on the production line of steel sheets. The presence of oil traces on the surface of steel sheets is believed to generate complex mechanical relaxation spectra in the temperature range 180K-300K, as reported in the literature [1]. Actually, the mechanisms responsible are not completely understood.

In order to elucidate this question, mechanical spectroscopy measurements were performed on cold-rolled steel sheets with different surface states, together with measurements on nearly freely supported oil films.

2. Materials and techniques

2.1 Materials

Three kinds of steel sheets (% composition: 0.02C, 0.23Mn, 0.02Si, 0.008P, 0.01S, 0.04Cr, 0.035Al, 0.0039N , 0.02Cu and Fe balance) were tested, namely:
(1) samples in the as received state (*i.e*. after cold rolling and industrial cleaning)
(2) laboratory cleaned samples (the surface layer was cleaned and chemically etched)
(3) clean samples (as in (2)) and covered with a thin oil film.
In addition, experiments with thin cellulose substrate saturated with oil were carried out in order to access the behavior of oil alone.

Oils with different amount of sulphur (0.40 to 0.70 %) were tested. Only results related to oil containing 0.40% sulphur are reported in this paper. According to InfraRed spectroscopy, the oils were mainly groundnuts-based [1].

2.2 Techniques

High resolution mechanical spectroscopy experiments were carried out using two complementary types of spectrometers: (*i*) an inverted pendulum working in the resonant mode (around f=1Hz) and (*ii*) a low frequency spectrometer operated in the forced oscillation mode. In the case of both instruments, the specimens were tested in torsion thus allowing measuring the torsion stiffness K with a very low (10^{-4} to 10^{-6}) relative strain amplitude. K is proportional to the shear modulus G in the case of isotropic and homogeneous material.

The first type of spectrometer allows measuring the variations of the shear modulus G (proportional to f^2) and the loss factor $Q^{-1}=\delta/2\pi$ (δ is the log decrement of the free decay oscillations) of the specimen *vs* temperature. The low frequency spectrometer makes it possible to measure the dynamic shear modulus $G^* = G'+jG'' = G'(1+j\tan\varphi)$ by both temperature (90K to 470K) and frequency (10^{-4}Hz to 5Hz) scanning, as described elsewhere [2]. In the case of low loss values, $\tan\varphi$ is close to Q^{-1}.

3. Results

The behavior of steel specimens and of oil was measured using the first and second type of spectrometers, respectively.

Mechanical loss spectra Q^{-1} (T) measured at nearly constant frequency (around 1.3Hz) for the as-received sample and for the laboratory cleaned sample as shown on Fig. 1. It is obvious that phenomena displayed by the as-received sample below 300K are attributed to surface effects. Mechanical loss spectra (Fig. 2) taken on samples covered with thin oil film after laboratory cleaning, clearly confirm that oil itself is responsible of these low temperature phenomena.

In order study in detail the behavior of oil alone, specific specimens were prepared using thin (thickness 90 μm) cellulose substrates exhibiting negligible stiffness. These substrates were saturated with the oil to be investigated. Mechanical spectroscopy of oil was then performed by temperature scanning at several frequencies (Fig. 3). Three relaxation processes are to be observed below 300K, at nearly 190K, 215K and 270K, called processes O1, O2 and O3, respectively. It is observed that the shear modulus of oil at very low temperature (*i.e*. below 175K) is about 2.5 GPa, which is a classical value for organic material in the solid state [3].

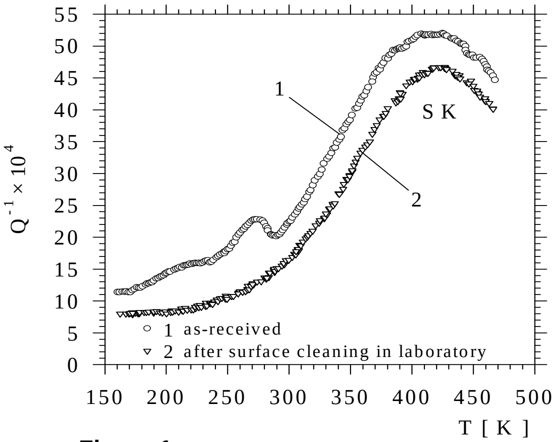

Figure 1:
Mechanical loss spectra of cold-rolled steel sheet.
Curve 1: as received state
Curve 2: after laboratory cleaning.
SK is the Snoek-Köster process exhibited by the steel specimen.

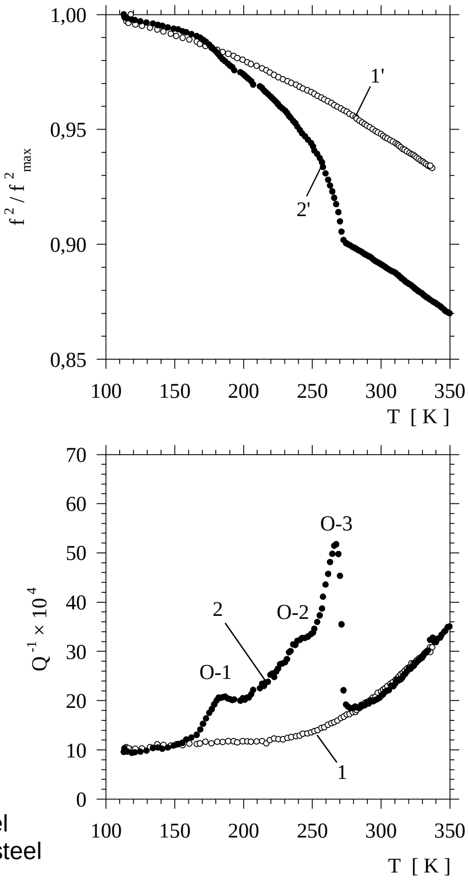

Figure 2:
Low temperature relaxation peaks (2) and normalized modulus (2') observed after a thin film oil deposition on a laboratory cleaned steel sheet (curves 1 and 1' are related to cleaned steel sheet before oil deposition).

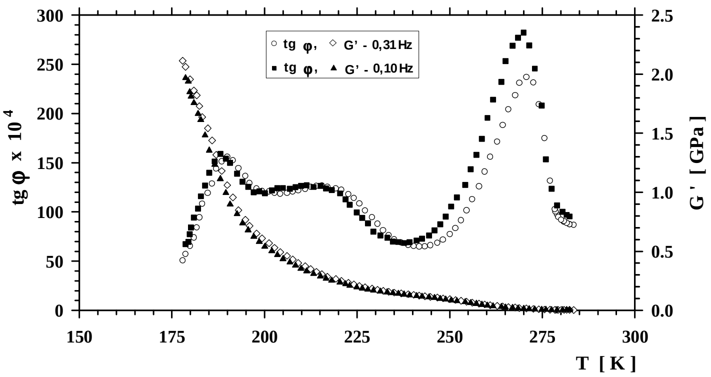

Figure 3: Mechanical loss factor tanφ and real part *G*' of shear modulus of oil (0.40% sulphur) as a function of temperature measured at two frequencies.

The shear modulus drastically decreases due to O1 relaxation process. Process O3 is characterized by a strongly asymmetrical loss peak, the high temperature side of which appearing non-thermally activated. This aspect is typical of a phase transition and could be attributed to the melting of some water content in oil. High resolution low frequency mechanical spectroscopy makes it possible to obtain the characteristics (apparent activation enthalpy ΔH and preexponential factor τ_0) of the relaxation processes, in particular process O1, as reported in Table 1. The preexponential factor τ_0 is extremely low, far below the expected value (of the order of 10^{-13}s) for a classical relaxation process. Obviously, process O1 is non Arrhenian. Such low values of τ_0 are generally associated with cooperative atomic motions or rearrangements like those involved in a glass transition phenomenon. Actually, the drastic decrease of modulus is in agreement with such an assumption.

Table 1: characteristics of O1 oil relaxation process

Sulphur content (%)	ΔH (eV)	τ_0 (s)
0.40	1.33	$3.76\ 10^{-36}$
0.41	1.34	$2.69\ 10^{-36}$
0.74	1.56	$3.74\ 10^{-41}$

The observations reported above indicate that the oil film is responsible for low temperature relaxation effects. The comparison of the mechanical loss spectra exhibited by (*i*) oil alone and (*ii*) a steel sheet laboratory cleaned and then covered with a thin oil film (Figs. 2,3) supports this assumption. Obviously, the low temperature relaxation processes exhibited by the steel sheet and oil have similar aspects and occur in the same temperature ranges.

4. Discussion and conclusion

The low temperature mechanical loss peaks observed in metallic [4-6] and ceramic [6] substrates coated with thin films of synthetic oil or grease have been interpreted in terms of hydrogen-induced relaxation effects occurring in a metallic substrate. On the contrary, the results reported here suggest that oil itself is responsible for these three processes: glass transition of the amorphous phase (O1 process), relaxation and melting of a water-rich phase (process O3). The origin of O2 relaxation process remains unclear.

It is shown that high resolution mechanical spectroscopy is a very sensitive technique to detect the presence of extremely fine oil film on the surface of substrate and particularly oil debris remaining on the surface of cold-rolled steel sheets.

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