

# Impedance analysis of Portevin-Le Chatelier effect on aluminium alloy

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## Abstract

The paper presents the results of an electrochemical investigation of the surface PLC effect connected with a non-uniform process of plastic flow of aluminium alloys containing magnesium. The PLC effect exemplified as a serration results in a non-homogenous change of an external surface of a stressed sample. The dynamic electrochemical impedance spectroscopy investigations were carried out in dynamic conditions during tensile tests. The surface PLC effect resulted in cyclic changes of dc current values and of instantaneous impedance spectra, which were correlated with the oscillatory character of measured mechanical parameters. An analysis with the electrical equivalent circuit revealed that the process discussed was a multistage one, depended on polarization and was directly connected with mechanical factors of the PLC effect. The most distinct surface PLC effect was found for the low levels of anodic polarization, where a tendency to repassivation is low. For high polarization a number of phenomena occur simultaneously, such as the repassivation processes and the initiation of local corrosion, which cause significant problems in an analysis of these studies. © 2006 Elsevier Ltd. All rights reserved.

**Keywords:** Portevin-Le Chatelier effect; DEIS; Al–Mg alloys

## 1. Introduction

The Portevin-Le Chatelier (PLC) effect reveals as a serration due to a load applied or as abrupt elongation during plastic deformation of many alloys. This effect is influenced by temperature, strain rate and pretreatment. It is the most evident example of plastic instability of alloys. The theoretical fundamentals of the PLC effect were developed by Cottrell [1,2], who presented an idea of “Dynamic Strain Aging” (DSA). A deformation is described by the total strain  $\varepsilon_{TOT}$ , which is a sum of the elastic strain  $\varepsilon_E$  and the plastic strain  $\varepsilon_P$ . The elastic strain is totally reversible and is related to the Young’s modulus and the stress applied by the Hooke’s law:

$$\sigma = E\varepsilon_E \quad (1)$$

The plastic strain is non-reversible and is caused by the load applied. In a range of low stresses the strain is described by an exponential dependence and is called a creep. The value of stress, for which the strain occurs is termed as a yield stress  $\sigma_F$ . In many cases this quantity depends on a square root of the plastic strain

$\varepsilon_P$ . The yield stress is also influenced by the strain rate. It is so due to an interaction between moving dislocations and a diffusion of contaminating elements. The dependency between of the yield stress and the strain rate is also named a strain-rate sensitiveness. Generally, it is accepted that the rate of plastic strain depends exponentially on a difference between the stress applied and the yield stress [3–5]:

$$\frac{d\varepsilon_P}{dt} = \dot{\varepsilon}_P = \frac{1}{\tau_1} \exp \left[ \frac{\sigma - \sigma_F(\varepsilon_P, \dot{\varepsilon}_P)}{E} \right] \quad (2)$$

where  $E$  is the Young’s modulus,  $\tau_1$  the time constant,  $\sigma$  the stress,  $\sigma_F$  the yield stress,  $\varepsilon_P$  the plastic strain, and  $d\varepsilon_P/dt$  is the plastic strain rate.

A phenomenological description of the Portevin-Le Chatelier effect stems from an observation that the yield stress is not constant, but depends on the strain rate. A homogenous deformation is unstable when the yield stress decreases with an increase in the strain rate. This behaviour is related to negative strain-rate sensitiveness and results in instability exhibited as the serrations on a stress–strain curve [6]. Precise and unequivocal dependence between the yield stress and the strain rate is not known, so an interpretation of the experimental data is troublesome. There are two basic groups of the experiments carried out. Bodner and Rosen [7,8] applied constant strain rate during a tensile test and

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measured the stress necessary to obtain a defined strain. They concluded that lower stress was needed when the strain rate was higher. In the experiments done by van den Brink et al. [9] and Linga et al. [10] the specimens deformed were subjected to abrupt changes of the strain rate. The strain-rate sensitiveness  $\Delta\sigma/\Delta(d\varepsilon_p/dt)$  was determined by a measurement of the stress changes  $\Delta\sigma$  accompanying the strain rate changes  $\Delta(d\varepsilon_p/dt)$ . For certain range of strain  $\varepsilon_p$  and strain rate  $d\varepsilon_p/dt$  the corresponding stress was lower than initially. In other words, for high strain rates the target deformations were attained with lower stress. On the other hand, for low strain the strain-rate sensitiveness was positive. A detailed description of this complex phenomenon was presented in the review papers by Estrin and Kubin [11] as well as by Franklin et al. [12].

The Portevin-Le Chatelier effect is interesting not only from the scientific point of view, but also due to some practical reasons. The serration is revealed by many alloys, including Al–Mg ones, which are widely applied in practice. Unfortunately, the Al–Mg alloys undergo corrosion accelerated by mechanical factors. Stress-corrosion cracking (SCC) of aluminium alloys is typically intergranular. According to the electrochemical theory this requires conditions along grain boundaries that make them anodic with respect to the rest of the microstructure so that corrosion propagates selectively along the boundaries. In the case of stress-corrosion cracking apart from the electrochemical also mechanical aspects play an important role. Whether or not the SCC develops in susceptible aluminium alloy product depends on both magnitude and duration of tensile stress acting on the surface.

Plastic deformation of Al–Mg alloys is often accompanied by plastic instabilities due to the DSA and the interaction between dislocations [13–17].

The serration develops at the grain boundaries of Al–Mg as a result of local migrations of the dislocations and contaminating elements. In the alloys with limited dynamic recovery, the serration is also related to an initiation of dynamic recrystallization, which is very important in preventing the progress of grain boundary cracking [18,19]. The serrated flow of aluminium alloys was investigated by Tian [20] and Close et al. [21]. They found different types of the serration. However, no corresponding change of types of the serration and characteristics of stress drop distributions with flow stress was found. The experimental results obtained on Al–Mg alloys by Horwath et al. [22] showed that the occurrence and development of the plastic instabilities – serrated indentation – depend strongly on the solute content. During the dynamic microindentation tests the amplitude of microhardness was changing as a power law function of Mg solute content with an exponent of 2/3. Simultaneously, a kinetics of Mg solute segregation during the DSA was controlled by the pipe diffusion. In order to elucidate the shape of serration and its mechanisms a lot of models with computer simulations were proposed [23–31]. The Portevin-Le Chatelier effect has another practical implications. The serrated flow and associated progressive surface markings severely restrict the application of some aluminium sheet alloys for automotive body exteriors [32]. Self-organized dislocation structures in alloys are directly connected with fatigue damage [33–35].

Traditionally, the Portevin-Le Chatelier effect was investigated in details by means of mechanical test methods [22,36–45], acoustic emission [24,45–48], scanning electron microscopy [49–51], a novel microscopic strain mapping technique [52].

However, in literature there are no information about electrochemical investigations of this interesting effect as regards practical and scientific point of view. The serrated flow of dislocations and physico-chemical changes of surface have to determine the changes of electrode impedance. In other words, the serration process is not stationary, therefore the classical “frequency by frequency” impedance measurements are not suitable. In this case the dynamic electrochemical impedance spectroscopy (DEIS) may be successfully applied [53–56]. For electrochemical description of the Portevin-Le Chatelier effect the serration process dynamics versus electrode potential is required. There arise some important questions: What is the influence of relaxation changes of strain on electrochemical processes? In other words, what is the dependency between the mechanical and electrochemical PLC effect? This paper is devoted to these problems. The experiments were performed on the aluminium alloy A9 5251, which is widely applied in practice. Moreover, it is the material revealing distinct mechanical PLC effect [57].

## 2. Experimental

Detailed electrochemical measurements during the tensile test were conducted for the A9 5251 aluminium alloy. The samples were mounted in a measurement cell and then in the tensile testing machine MTS 81012. Tensile tests and samples preparations were performed according to the EN 10002-1 + AC1 standard at room temperature with the MTS extensometer characterized by the measurement base of 40 mm. The examinations were carried out in a three-electrode electrochemical cell in which silver/silver chloride electrode was used as a reference electrode and the auxiliary electrode was made of platinum net. The measurements were performed in buffered solution ( $\text{H}_3\text{BO}_3 + \text{Na}_3\text{BO}_3$ ; pH 7.4) with additional content of 0.002 M sodium chloride.

The strain rate was equal to  $0.005 \text{ s}^{-1}$  (according to the standard EN 10002-1 + AC1). Additionally, the electrochemical examinations were carried out under tensile stresses in such a way that time of the DEIS measurements was correlated with the strain process. Total measurement time was equal to 278 s. The measurement had been stopped when the sample was broken. The maximum recorded strain value for the A9 5251 was of 18% (0.18).

The measurement set-up consisted of the electrochemical system: a potentiostat and a National Instruments PCI 6120 card generating the perturbation signal, registering voltage perturbation and current response signals. Impedance measurements were carried out for the frequency range: 20 kHz–7 Hz. The sampling frequency of 50 kHz resulted from the settings of measurement card being at our disposal. In our investigations the multi-sinusoidal perturbation signal composed of 20 sinusoids was utilized.

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