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Experimental and numerical investigation on carbonitrided steel characterization with spherical indentation



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ABSTRACT

This study investigates the mechanical behavior of a carbonitrided steel, plastically graded material, during a spherical indentation test. We demonstrate that the behavior of a carbonitrided steel can be simulated accurately with a seven layer sample, including the surface, five intermediate layers and the substrate. Moreover, for engineering design simulations which require a lot of calculation time, using three layers provides a good compromise for accuracy.

A complete experimental procedure involving seven successive identifications with inverse analysis gave the same results as a simpler procedure proposed in a previous paper. It is shown that the assumption of a linear variation of the properties in the intermediate layers between surface and substrate is valid. This is the first time that an instrumented roller indentation test has demonstrated the reliability of the proposed method for the mechanical characterization of carbonitrided steels with spherical indentation.

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1. Introduction

Surface heat treatment such as carbonitriding is often used to combine high surface strength and high core toughness of mechanical parts submitted to wear and fatigue. The carbonitrided steels present a variation in hardness with depth from the surface while the elastic properties are not affected by the treatment and are constants with depth [1–4]. For these reasons they can be called plastically graded materials (PGMs).

The use of classic tensile tests on that kind of material does not provide an accurate prediction of the variation of the mechanical properties with depth. Indeed, carbonitriding alters only the top layers of the material, which have a very small volume compared to the volume of the whole part. However, mechanical characterization of PGMs is an important challenge for engineering industries. A precise knowledge of the mechanical properties is necessary to obtain efficient finite element (FE) results for the design of engineering parts. Considering this problem, instrumented indentation tests have a great potential for the characterization of PGMs due to the small volume of material tested.

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It has been demonstrated that the indentation test can provide an accurate prediction of the mechanical behavior of homogenous materials [5–14]. This test does not directly provide the stress–strain curve $\sigma(\varepsilon)$ of the tested sample but an indentation load–depth curve F(h) from which the parameters of the material's hardening law can be determined. The simpler and quite accurate elastic–plastic law for metals is the piecewise linear/Hollomon's power-law [15,16] given by Eq. (1):

$$\begin{cases} \sigma = E\varepsilon; \sigma < \sigma_y \\ \sigma = \sigma_y^{1-n} E^n \varepsilon^n; \sigma \ge \sigma_y \end{cases}$$
(1)

where *E* is the Young's modulus, σ_y the yield stress and *n* the strain hardening exponent.

The determination of the mechanical parameters of Eq. (1) from the indentation curve can be undertaken using multiple approaches. The first one is often called "reverse analysis" and is based on the minimization of the least squares deviation between indentation experiments and models, linking the indentation load, the displacement and the hardening law parameters, previously determined by numerical simulations [5–10]. The second approach is called "inverse analysis" where the minimization is directly carried out between experiments and FE simulations. Several authors [11–13] suggested applying the inverse analysis approach on indentation experiments in order to determine some mechanical properties. Using "reverse analysis" with

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predetermined models of indentation is convenient because it does not require additional FE simulations and thus is a quick method. However, the proposed models of indentation data are limited to a great number of conditions, such as indenter shape, mechanical behavior of the tested sample, homogeneity of the sample, and friction conditions. For this reason "inverse analysis" is a good alternative method when the conditions of the test are not taken into account in the proposed models of indentation, as is done for more complex behavior laws [12,13]. The third approach [14] consists of building up a database of FE simulation curves and calculating the error between the experimental curve and the curves of the database. The definition of the equation that describes the distribution of the error allows for the determination of the parameters of the hardening law that represents the material.

A complete overview of the theory realized by Moussa et al. [13] has shown that, compared to homogenous materials, few studies have been done to characterize the mechanical behavior of PGMs with indentation testing. These studies can be divided into two groups. The first one consists of performing many tests in the sectioned part of the sample [17-20]. The second one consists of making only one indentation test at the surface and then applying a mixture law [1,3,21–32]. Moussa et al. [13] showed that these methods present many drawbacks for the case of carbonitrided steels. Because of these drawbacks, Moussa et al. [13] developed a method to characterize the mechanical behavior of carbonitrided steels using spherical indentation and the inverse analysis. This method consists, in a first step, to characterize the substrate as a homogenous material since its volume is large enough. With the assumption that the variation of the hardening law in the carbonitrided layers is linear, the corresponding hardening laws are determined in a second step.

In the present paper, a carbonitrided C12 steel is characterized with a method based on no assumption about the evolution of the hardening law with depth. The results obtained with the two methods are compared in order to verify the validity of the assumption of linear evolution of the hardening law with depth. Finally, a validation test is proposed in order to demonstrate the reliability of the proposed method for the mechanical characterization of the carbonitrided steels with spherical indentation.

2. Material presentation: hardness profile and metallographic analysis

In the previous paper, we studied two C18 carbonitrided steels [13]. In this paper, we propose to study a C12 carbonitrided steel (0.12% of carbon before the treatment). The carbonitriding treatment was carried out at Faurecia Automotive Seating, however, details of the treatment cannot be outlined due to confidentiality restrictions. The hardness profiles of this steel, obtained on three different samples, are presented in Fig. 1(a). The hardness profile of the C12 carbonitrided steel can be described as follows. The hardness is approximately constant in an outer layer of thickness e1, then, the hardness decreases steadily in an intermediate zone of thickness e₂. Finally, for a depth greater than $e_1 + e_2$, the hardness is constant in a third zone which corresponds to the substrate. Fig. 1 shows that the hardness can be approximated by a linear function in the intermediate layers (Fig. 1(a)). In the proposed procedure, the values of e₁ and e₂ were obtained from the minimization of least squares deviation between the experimental hardness profile and the one approximated with three segments (see Fig. 1(a)). For the studied material, the thickness is $e_1 =$ 0.06 mm for the outer layer and is $e_2 = 0.29$ mm for the intermediate lavers.

In order to analyze the variation of the microstructure in the intermediate layers, the outer parts of the C12 carbonitrided samples were removed by rectification and polishing. After the polishing process using fine emery papers (up to 1200 grit) and an electrolytic polishing machine, nital etchant was used to reveal the microstructure.



Fig. 1. (a) Hardness profile of the carbonitrided C12 steel obtained from three samples. (b) Simplified hardening profile with the depths of the samples used in the characterization.

The microstructures of the carbonitrided C12 steel for different depths are presented in Fig. 2 and their positions on the hardness profile are shown in Fig. 1(a). Fig. 2(a) shows the microstructure of the surface of the carbonitrided C12 steel in which very fine needles of martensite can be observed. In Fig. 2(b), the microstructure consists of a martensite or bainite matrix containing troostite particles in the former Austenite grain boundaries. In this figure, the presence of small grains of ferrite can also be observed. The microstructure of the substrate, which contains acicular ferrite grains and pearlite, is given in Fig. 2(c). The evolution in the depth of the microstructure demonstrates, as a consequence, changes in the plastic properties of the material that can be observed in the hardness profile given in Fig. 1.

3. Experimental conditions, finite element model and inverse analysis

The experimental indentation tests were conducted using an inhouse instrumented indentation bench. A spherical tungsten carbide indenter of 1 mm diameter was used. The Young's modulus and the Poisson's ratio of the indenter are E = 600 GPa and $\nu = 0.23$ respectively. The indentation load was led up to 900 N with a constant

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