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Prediction of viscoplastic properties of polymeric materials using sharp indentation

Noriyuki Inoue^a, Akio Yonezu^{a,}*, Yousuke Watanabe^a, Takeo Okamura ^b, Kouji Yoneda ^b, Baoxing Xu ^{c,*}

a Department of Precision Mechanics, Chuo University, 1-13-27 Kasuga, Bunkyo, Tokyo 112-8551, Japan

^b Material Analysis Department, NISSAN ARC, Ltd., 1 Natsushima-cho, Yokosuka, Kanagawa 237-0061, Japan ^c Department of Mechanical and Aerospace Engineering, University of Virginia, Charlottesville, VA 22904, USA

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A B S T R A C T

The present study proposes a new indentation method to predict the viscoplastic properties of polymeric materials utilizing three different indentation experiments. Numerical experiments with finite element method (FEM) are first carried out to simulate response of materials with various elastic and viscoplastic properties. The strain rate is featured by a loading rate of indentation, and the hardening rate can be captured through variations of the indenter shape. Next, a parametric FEM study is conducted to build the relationship between indentation load-depth curves and material parameters. Dimensionless analysis is employed to represent the relationship with simple formulae, through which, a reverse algorithm is proposed to extract the viscoplastic properties. Finally, both numerical and experimental investigations are performed to verify the proposed method.

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1. Introduction

The ever-increasing use of polymeric materials with a small volume, such as coatings to achieve a desirable surface properties and thin substrates to achieve a high flexibility of wearable devices, requires appropriate characterization of their mechanical properties down to the nanoscale. Usually, almost all of polymeric materials feature time-dependent mechanical properties and in particular, the plastic properties (often referred to as viscoplasticity) are of interests to the researchers. Given the limits on their volume and scale in applications, the conventional tensile or compressive testing technique is very challenging in the accurate prediction of their mechanical properties. Instrumented indentation technique provides a compelling alternative with the merits of almost no requirements in both volume and scale of samples and accuracy in measurements, and has been widely employed to probe the mechanical properties like Young's modulus and hardness [\[1,2\]](#page--1-0). Elastoplastic properties have been successfully derived from curves of indentation force and penetration depth and impressions (i.e. residual imprints) $[3-5]$. In these studies, the dimensionless function that correlates the parameters of indentation responses (mostly the indentation curves) with the elastoplastic properties of materials is first established through forward analysis (i.e. the acquirement of indentation responses from serials of assumed mechanical parameters of materials) using computational approaches $[6-9]$. The elastoplastic properties of unknown materials can then be identified when the indentation responses obtained from practical experiments are assigned to the dimensionless function, which is a reverse analysis process (i.e. identification of material properties from indentation responses). To our knowledge, the reverse analysis may have a higher resolution than that of conventional analytical solution (i.e. hardness measurement and Tabor equation), because it does not require on the accurate estimation of indentation contact areas, and can use a robust representative strain [\[10,11\].](#page--1-0) Therefore, the instrumented indentation based on the reverse analysis has been acknowledged in the determination of the elastoplastic properties of materials.

The evaluations of time-dependent mechanical properties of materials such as polymeric materials through indentation are mostly focused on viscoelasticities [\[12,13\]](#page--1-0) and there are only a few of measurements on viscoplastic properties so far. As one of pioneering work, Bucaille et al. employed nanoindentation test to evaluate viscoplastic properties of polycarbonaite (PC) with finite element method (FEM) [\[14\].](#page--1-0) By matching the computational and experimental curves with the help of various FEM computations, they could quantitatively predict the stress–strain curve at large plastic deformation. However, their method is a typical forward analysis and relies on intense FEM computations. Kermouche et al. employed an analytical approximate solution by using

[⇑] Corresponding authors. Tel.: +81 3 3817 1829; fax: +81 3 3817 1820 (A. Yonezu). Tel.: +1 434 924 1038; fax: +1 434 982 2037 (B. Xu).

E-mail addresses: yonezu@mech.chuo-u.ac.jp (A. Yonezu), bx4c@virginia.edu (B. Xu).

classical indentation parameters (e.g. mean pressure, indentation strain rate and representative stress–strain) and developed a method for evaluating viscoplastic properties of glassy polymer [\[15\]](#page--1-0). Their method is relatively simple in comparison of the forward analysis and is expected to be applied to a wide range of materials. For robust estimations of viscoplastic properties of polymeric materials, dimensionless functions (established by parametric FEM study preliminarily) may be more powerful for exploring a simple, convenient and accurate approach and is worth of investigation. More recently, Peng et al. used dimensionless function to estimate the yield stress of polymeric materials (PVC) with an assumption of viscoelastic-perfectly plastic material [\[16\].](#page--1-0) However, it did not focus on the flow stress and non-linear work hardening behavior.

The present study is establishing a new method to evaluate viscoplastic properties using an indentation method with the help of dimensional analysis. To focus on the study of viscoplasticity in indentation experiments, we employ a polymer material – polycarbonaite (PC) which has little viscoelasticity yet a very strong viscoplasticity with non-linear work hardening behavior. The present method aims to predict a constitutive law of stress–strain relationship up to a large plastic strain with the consideration of the strain rate effect. The process of estimation employs a reverse analysis based on the data of indentation loading curve, and three indentation experiments conducted using two sharp indenters with different angles and loading rates. First, numerical experiments with finite element method (FEM) are carried out in order to simulate indentation loading curves against the materials with various viscoplastic properties. Next, a dimensionless function which can correlate the materials properties with the indentation loading curves is established in the reverse analysis. Finally, the proposed method is verified through numerical and experimental results.

2. Materials and the mechanical constitutive law

Three types of polymeric materials, engineering glassy polymer materials, polycarbonate PC, polymethylmethacrylate PMMA, and acrylonitrile butadiene styrene ABS, are investigated in this study. Fig. 1 shows their typical compressive stress–strain curves. These tests were conducted in our laboratory (details please see Section [5.2\)](#page--1-0). In Fig. 1, during the elastic deformation, all of their stress–strain relationships are almost linear. At the yield point, the stress shows a peak, and then slightly decreases when plastic deformation occurs. Plastic flow stress remains almost constant at first, and increases beyond a critical plastic strain. This behavior is well known to be dependent on strain rate. A high strain rate will promote the stress, whereas a low strain rate will decreases the

Fig. 1. True stress–true strain behavior in uniaxial compression test for PC, PMMA, and ABS at strain rate of 10^{-4} s $^{-1}$.

Fig. 2. True stress-true strain behavior in uniaxial compression test for PC at strain rate of 10^{-3} , 10^{-4} and 10^{-5} s⁻¹.

flow stress. Fig. 2 shows the stress–strain curves of PC with different strain rates. The duration of plastic deformation strongly depends on the strain rate, yet not for the elastic deformation. This study will focus on plastic deformation with strain rate effect.

This non-linear plastic deformation can be approximately described by fitting their corresponding curves of stress–plastic strain, as shown in Fig. 2 and the full plastic deformation can be expressed by the following constitutive law.

$$
\sigma = K \cdot e^{h_g \bar{e}_p^2} \cdot \dot{\bar{\epsilon}}^m \tag{1}
$$

In Eq. (1), ε_p is the plastic strain and $\dot{\bar{\varepsilon}}$ is the strain rate. Note that this study ignores stress softening phenomena around the yield point (i.e. peak stress) of relevance to changes of molecular structure. This simplification is developed from the G'sell-Jonas law [\[17\]](#page--1-0), and has been used by Kermouche et al. [\[15\].](#page--1-0) and Bucaille et al. $[14]$. In addition, the materials constant, m represents the strain rate sensitivity, K is the consistency parameter, and h_g is the strain hardening modulus. These three material constants will be identified from indentation experiments in this study. In Fig. 2, three curves with different strain rates ($\bar{\varepsilon} = 10^{-3} - 10^{-5}$) can be fitted well by Eq. (1) , and the resultant materials constants are listed in Table 1. Similarly, the other materials PMMA and ABS are investigated and their material parameters are also included in Table 1. Note that elastic property (Young's modulus E and Poisson's ratio v) is from the previous studies $[14,15,18]$ and is assumed to be known in advance.

3. Numerical analysis

3.1. FE model

[Fig. 3](#page--1-0) shows the FE model of an indentation test with a conical sharp indenter. This study uses two types of conical indenters with different apex angles. The sharper angle of indenter is called ''Indenter A", whose half apex angle θ is 30°, and the other "Indenter B" has θ = 70.3°. These indenters are employed to mimic triangle pyramidal indenters, whose diagonal angle α is 70 \degree for "Indenter

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