Microelectronics Reliability 74 (2017) 121–130

Contents lists available at ScienceDirect

Microelectronics Reliability

journal homepage: www.elsevier.com/locate/microrel

Viscoplastic properties of pressure-less sintered silver materials using indentation

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ARTICLE INFO

Article history:
Received 15 November 2016
Received in revised form 19 February 2017
Accepted 10 April 2017
Available online 25 April 2017

Keywords:
Indentation
Viscoplastic
Sinter
Porous

ABSTRACT

This paper focuses on using a combination of indentation and FEA methods for characterizing the viscoplastic behavior of heterogeneous materials which pose additional challenges because of the non-uniform morphology. In particular, the paper focuses on two forms of pressure-less sintered silver interconnect materials: an adhesive-based particulate composite for low temperature applications and a porous sintered version for high-temperature applications. By using two different post-processing methods (an analytic approach and a computational FEA approach) for the indentation results, we obtain lower and upper estimates to the viscoplastic properties for both of these heterogeneous morphologies. Two types of indenters, spherical and Berkovich, and two types of indentation tests, constant load and constant strain rate, are compared, with regard to their ability to measure the viscoplastic properties of heterogeneous materials.

1. Introduction

Indentation techniques for extracting sub-surface properties of materials have been in use since the early 1900s. The vast majority of indentation tests were utilized to extract surface hardness properties, using indenters of known material and geometry. As commercial products miniaturized, the length scale for test methods have followed suit. Accordingly, nanoindentation capability was introduced in the early 1970s for studying material behavior at extremely small length scales.

Test methodology and the process of extracting hardness and elastic behavior for homogenous material using indentation is well understood [1,2,3,4]. The methodology for extracting the elastic modulus and hardness from indentation tests, without imaging of the indentation sites, stretch back to Doerner and Nix's work in 1986 for sharp indentation [1]. Comprehensive work can be attributed to Oliver and Pharr, who continued to refine these methods to include the effect of material pile-up and sink-in on the projected indentation area [2]. Additional work was done by Giannakopoulos and Suresh in studying inelastic behavior using indentation with sharp indenters like Berkovich indenters [3], and Alcalá et al. did similar work for spherical indenters [4].

More recent nanoindentation studies have also focused on characterization of viscoplastic behavior [5,6,7,8,9]. Lucas and Oliver in 1999 described several indentation tests while studying high-purity indium [5]. Among those tests, they were the first describe a method to obtain a constant 'indentation strain rate' by controlling the loading rate such that the loading rate divided by the load (which will be referred to as the normalized load rate) remained a constant. Tests maintaining a constant normalized load rate (NLR) are referred to in indentation literature as a constant strain rate test [6]. More details on this test and the term 'indentation strain rate' is explained in more detail in Section 2, because it is defined differently than strain rate is in uniaxial tests for bulk specimens.

Constant force tests using indentation has been in use for a longer time [7,8]. Mayo et al. used constant force tests on TiO to extract the hardness, elastic modulus, and strain rate sensitivity [7]. Mayo's tests consisted of using a loading rate to get to a prescribed depth and then holding the load constant. The present study uses a high loading rate to reach the desired constant load for the test, but there is no predetermined depth. A more recent use of the constant force test can be seen in Hasnine et al.'s work on SAC305 (96.5% tin, 3% silver, and 0.5% copper) solder [9]. Hasnine described a procedure to extract the entire creep curve from a single constant force test. Yet there have been several studies that have shown an instability in the steady-state region of the constant load test which affects the extracted creep exponent.

Peykov et al.'s study compared results from constant force tests with those from constant indentation strain rate tests [6]. Peykov found that the steady-state regions of the constant load tests showed increasingly high strain rate sensitivity values with decreasing loads while those from the constant indentation strain rate tests remained constant. He attributed this rise to thermal drift error occurring during the steady-state region being comparable to the penetration depth and suggested the use of increased force. Dean et al. in 2014 [10], performed a comprehensive study on steady-state instability. Dean found that the extracted creep exponent was much larger for many constant force test variants than found in conventional uniaxial testing of bulk specimens. The extracted creep exponent varied quite widely depending on indenter

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http://dx.doi.org/10.1016/j.microrel.2017.04.009
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type, maximum load level, and ramp rate to maximum load level. He ascribed the instability to two possible factors: (i) primary creep still being active in a significant portion of the indent area even during what is considered the steady-state region; and (ii) the empirical models used in indentation literature may be overestimating the "equivalent" stress and strain rate. While making several suggestions to decrease the steady-state instability in the constant load tests, including larger indenters and higher force levels, Dean concludes that some form of numerical modeling is required. The present study attempts to mitigate this instability seen in Dean and Peykov's studies by using larger forces, larger diameter indenters (when applicable), and by using numerical analysis in conjunction with the constant load tests. On the numerical side, several studies have described methodologies to correct empirically estimated material parameters by using FEA modeling to post-process the test results. Hamasaki et al. and Roshanghias et al. used inverse iteration methods that estimate material properties by fitting FEA simulation results to experimentally obtained P-h (load-displacement) curves [11,12].

Use of indentation methods for the characterization of mechanical properties of sintered materials poses additional challenges because of their complex porous heterogeneous morphology [13,14,15,16]. In 1991, Fleck et al. studied the effect of porosity on indentation results [13]. Fleck used the Gurson model for modeling low porosity materials and a particle yield model for larger porosities. In 2006, Chen et al. proposed a new technique for extracting elastic-plastic properties of porous films using spherical indenters [14]. Chen used FEA in conjunction with Gurson's model to account for the densification that occurs during an indentation. He found that the densification affected the extracted hardness, but had negligible effects on the modulus. Recently Chen et al. in 2016 used this same approach of combining FEA with Gurson's model to investigate the properties of LCSF ($La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$) with variable porosities (5–45%) [16].

Utilizing indentation for extracting viscoplastic properties for porous materials, while less common in literature, has seen use in investigations of porous films and bones [17,18,19,20]. Padmanabhan et al. in 2005 [17], studied the creep of porous nanocrystalline palladium using micro-indentation. Padmanabhan had measured the time-dependent microhardness variation. He et al.'s study in 2007 [18], showed the effect of the porosity on the creep deformation of hydroxyapatite, a possible bone substitute. He used the minimum solid area model to extract actual cross-sectional areas to show how increased porosity revealed increasing inelastic behavior for the brittle material. Zaki et al. in 2011 studied porous Cu-Ge ferrites with indentation [19]. From the high creep exponents, Zaki concluded that the dominant mechanism was dislocation creep. Zin et al. in 2012 used ball indention to describe the mechanical and thermal properties of porous low-dielectric films [20]. Among these, there is little literature to be found on the use of indentation to extract viscoplastic properties of silver sinters.

One important variable that can affect the extracted properties during indentation for many experiments is the ratio of the indentation depth, h, to material/film thickness, t. For thin films where the ratio h/t can be significant, the substrate below the thin film specimen can have an effect on the extracted elastic and plastic properties. As the specimen used in this study has a thickness of more than hundred times that of the maximum depth of the indents performed, the substrate effect is negligible in this study, so this extensive subject will only be covered briefly. There are two cases to consider, one where the substrate is harder than the film being indented into and vice versa. For the first case, Saha et al. found the effect on the extracted hardness to be negligible up to an h/t value of 1, penetration of the substrate, as long as the pile-up around the indenter was accounted for [21]. For the second case, Saha found that the true hardness of the film was only seen for h/t of <0.1 [21]. Furthermore, Sampath et al. found that at an h/t of 0.3 bending deformation in the film can occur and at an h/t of 0.5 membrane stresses are found in the film [22]. There are many models developed to extract the hardness of these film-substrate systems [23,24,25,26,27]. An early example is Bückle's use of a weighted sum equation to describe a two-layer system's hardness [23]. Later examples include models based on the law of mixtures using either the area of the indents [24] or the volumes of the plastic zones produced by the indent [25,26] in the film and substrate. For either case, if there is a significant mismatch of the elastic modulus of the film and substrate, the extracted modulus is affected by the complex interaction between them. Bückle in 1961 came up with the 10% rule for thick films (thicker than 500 nm), which stated that the elastic modulus can be extracted accurately for indent depths up to 10% of the film thickness [23]. There are also many models to derive the elastic properties of a thin-film system [1,28,29,30,31]. One important example is from 1968, when Doerner and Nix modeled the relationship empirically [1]. Doerner and Nix's work in this area has been improved upon by several groups since [28,29,30,31].

There have been many studies on methods to compare indentation and uniaxial test results for viscoplastic properties. Many studies compare these tests with similar forms of the power-law creep model, $\dot{\varepsilon} = A\sigma^n$ for the uniaxial tests and $\dot{\varepsilon}_i = B\sigma_i^m$ for the indentation tests. ε denotes the strain rate, σ is the stress, and the subscript i denotes an averaged value for these variables calculated from the indentation results which will be explained in detail in Section 3. The creep exponent found from indentation tests (m) has generally been in good agreement with that found during uniaxial creep tests (n), but the creep coefficients found in indentation tests (B) are generally larger than those from uniaxial tests (A) [32,33,34,35,36]. There have been many attempts at modeling this difference, one in particular is Bower et al.'s work [32]. Bower used finite element analysis to describe a relationship between coefficients A (uniaxial) and B (indentation). Bower quantified two parameters used in this relation: c, which accounted for the effect of pile-up ($c > 1$) and sink-in ($c < 1$) that occurs around the tip during indentation, and F(n), which was the reduced contact pressure as a function of the stress exponent. His analysis assumed a purely steady-state power-law creeping material with no transient creep. Su et al. used methods based on Bower's work to extract creep properties from amorphous selenium using indentation testing [33]. Su's simulations of constant force tests, which did not incorporate primary creep, demonstrated that the relative contribution of elastic deformations is initially very high (compared to creep deformation) and asymptotically decreases to a negligible value as the indentation depth increases. Su showed that this initial dominance of the elastic deformation increased the extracted creep exponent and affected the extracted creep coefficient unless the penetration depths are sufficiently large. Su provides some empirical guidance about the minimum required indentation depth. This effect will be discussed in Section 5 and in the conclusions.

Sintered silver is seen as an attractive material for use in power electronic devices as a die attach [37,38,39]. Sintered at relatively low temperatures of 200 to 300 °C, the finished product consists of a material with a melting point of 962 °C. Silver is also attractive due to its high electrical and thermal conductivity. Recently there have been sintered formulations that have been termed 'pressure-less' sintering by manufacturers. The term 'pressure-less' here implies that no additional ambient pressure is needed other than the self-weight of the components being bonded together. Pressure-less sintering provides a particularly attractive processing opportunity, since it requires less processing equipment (for adding additional pressure that sintering normally requires) and can be used with pressure-sensitive components. Yet there is a trade-off when it comes to a decrease in mechanical performance because of the increase in porosity [40,41]. Additionally, the strength of the interfacial bonding with the substrate can be compromised due to over-oxidation at the interface [42,43]. The interfacial issue can be overcome with addition of barrier layers. Two formulations for pressure-less sintered silver materials in the present study will include an adhesive-based particulate composite for low temperature applications and a porous version for high-temperature applications.