



**Journal of
Mechanics of
Materials and Structures**

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Volume 5, No. 2

February 2010

 **mathematical sciences publishers**

RATE DEPENDENCE OF INDENTATION SIZE EFFECTS IN FILLED SILICONE RUBBER

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Indentation experiments at the nanometer and micrometer range reported in the literature have shown that the deformation of polymers is — similarly to metals — size-dependent. In addition its size dependence, the deformation behavior of polymers is also known to be rate-dependent. Here silicone rubber is considered. In order to characterize the relation between size and rate-dependent deformation indentation tests of silicone at indentation depths of between 30 and 300 microns and loading times of between 1 and 1000 seconds are performed. In these experiments the hardness and dissipation increased with decreasing loading time and decreasing indentation depth. These experimental results are analyzed with a recently suggested indentation model which incorporates a Frank energy-related nonlocal deformation work. It is found that the indentation model is in good agreement with the experimental data. Evaluation of the experimental data indicates that the rate effects are mostly related to the nonlocal, size-dependent deformation.

1. Introduction

The indentation size effect manifests itself in increasing hardness at decreasing indentation depths and is fairly well investigated for metals [Ma and Clarke 1995; Nix and Gao 1998; Han et al. 2007a]. Such elevated hardnesses at small indentation depths have also been observed in polymers [Briscoe et al. 1998; Chong and Lam 1999; Tjernlund et al. 2004; Lim and Chaudhri 2006; Han and Nikolov 2007] but, in comparison to metals, for polymers these effects are not well understood. An understanding of size-dependent deformation behavior is important not only for small components in MEMS/NEMS (micro/nano-electromechanical systems), but also for the development of advanced material systems and composite materials with small material phases and other applications such as sealants, gaskets [Tan et al. 2007], and adhesives [Han et al. 2007b]. As many biological materials can be viewed as polymers, the relevance of size effects in polymers extends also to these materials [Yang et al. 2002].

Rate effects in indentation testing have been studied in [Sargent and Ashby 1992; Larsson and Carlsson 1998; Ebenstein and Wahl 2006; Tweedie and Van Vliet 2006], for instance, but without considering size effects. The effects of cross-linking and adhesion on the determination of the elasticity modulus of polydimethylsiloxane elastomers have been studied in [Carrillo et al. 2005]. A phenomenological constitutive model incorporating both rate and size effects has been suggested in [Gudmundson 2006], for instance. The elasticity modulus has also been determined to vary with the indentation depth [McFarland and Colton 2005; Tweedie et al. 2007]; this may be seen as a indication that a change in the dominant forces or mechanisms may be present at small indentation depths.

Keywords: indentation, hardness, polymeric material, viscoelasticity, size dependent deformation.

In [Han and Nikolov 2007] indentation experiments in the literature have been analyzed, and it has been found that the indentation size effect strongly depends on the polymeric material and its molecular structure [Han 2010]. For instance, Teflon (polytetrafluoroethylene) [Li and Bhushan 2000] does not seem to show any indentation size effects, while epoxy and polycarbonate show significant indentation size effects at indentation depths from 100 nm up to several microns [Chong and Lam 1999].

Assuming the rate of deformation does not alter the basic characteristics, these experimental results were evaluated with a hardness model suggested in [Han and Nikolov 2007]. The model and experimental results were found to be in good agreement for indentation depths above some 200 nm. The model is based on an elastoplastic extension of the rotation gradient model suggested in [Nikolov et al. 2007], which relates the length scale component of the deformation work to the Frank energy known from liquid crystal polymers. For indentation depths smaller than 200 nm surface effects may be present which are not represented in the hardness model. In metals such surface effects may include the effect of the curvature of the indenter tip [Qu et al. 2004], different deformation mechanisms close to free surfaces [Han et al. 2006], and surface roughness effects [Zhang and Xu 2002; Zhang et al. 2004].

Among the different polymers tested for size-dependent deformation, silicone rubber appears to exhibit astonishing behavior. While at the macroscale silicone rubber is very soft, at indentation depths of under one micron it exhibits higher hardness than many macroscopically harder polymers [Zhang and Xu 2002; Xu and Zhang 2004; Han and Nikolov 2007]. Here we investigate the rate dependence of the indentation size effect in silicone rubber with indentation tests at indentation depth ranges of approximately 30–300 microns and in loading time ranges of 1–1000 seconds. The data for these experiments are analyzed and discussed using the hardness model suggested in [Han and Nikolov 2007].

2. Experimental setting

Indentation system. The indentation tests were performed with the commercially available Fischerscope HM2000S indentation system (Fischer Technology Inc., USA). It is a load controlled system for the determination of hardness and other material properties [ISO 14577-1 2002]. The applicable load of the machine ranges between 0.4 and 2000 mN and the loading time between 0.6 and 10^6 seconds. The load and penetration depth resolutions are given by the manufacturer as $\leq 40 \mu\text{N}$ and $\leq 100 \text{ pm}$, respectively. Also according to the manufacturer the curvature of the indenter tip is guaranteed to be less than 500 nm, approximately between 200 and 300 nm; only polymeric materials with this indenter tip have been tested.

Silicone rubber material. The ingredients for the silicone rubber sample fabrication were obtained from Gelest, Inc., Morrisville, PA, USA. The silicone materials were produced in two parts, both of them silicone blends. Part A consists of fumed silica-reinforced vinyl-terminated polydimethylsiloxane (Gelest catalog code DMS-V31S15) as a base and a precious metal catalyst for vinyl-addition silicone cure platinum-divinyltetramethyldisiloxane complex in xylene (Gelest SIP 6831.2). Part B consist of vinyl-terminated polydimethylsiloxanes (Gelest DMS-V31) as vinyl silicone, methylhydrosiloxane-dimethylsiloxane copolymers and a trimethylsiloxy terminated (Gelest HMS-301) cross-linker.

For part A, 84.85 ml of the base was poured into a glass beaker by measuring its mass on a weighing machine; meanwhile, 3 ml of the catalyst was slowly added with a pipette. The compound was then thoroughly mixed with a stirrer for a couple of minutes. For part B, 80 ml of vinyl silicone and 20 ml of cross-linker were thoroughly mixed with a stirrer in another beaker. Parts A and B were poured in a

proportion of 3:1 into a polypropylene specimen cup. To get rid of any bubbles, the whole mixture was placed in an airtight chamber using a vacuum pump. The aggregate was then poured into a Petri dish (50 mm in diameter and 9 mm in depth, Pall Corporation, USA) to avoid damage and straining of the material due to handling. Finally the specimen was allowed to cure for several days at room temperature.

3. Indentation experiments

Figure 1 shows the indentation depth h and its projected counterpart h_c . Also shown are the elastic, W_I^e , and inelastic, W_I^p , parts of the total indentation work $W_I = W_I^e + W_I^p$ in the loading and unloading sequence of the load-displacement curve. A Berkovich indenter tip was applied with a characteristic angle of about $65^\circ 2'$. The nominal contact area is $A_s = c_B h^2$ for the Berkovich tip, where $c_B = 26.440$ [ISO 14577-1 2002].

The stress and strain fields are highly inhomogeneous during indentation [Dao et al. 2001]. Because of this inhomogeneity, strain fields and strain rates vary strongly. From a classical local continuum mechanics viewpoint, where the constitutive equations are described by the stresses and strains and their rates, these stress and strain fields should be affine to each other with h under certain conditions: first, the indenter tip geometry should be self-affine, that is, the nominal penetrating shape of the indenter tip should scale with the indentation depth, and, second, the strain and strain rate fields should also be affine to the indentation depth at a certain time.

For the self-affine cone geometry of the Berkovich indenter tip used here, the maximum indentation depth h_{max} is related to the maximal applied force F_{max} by

$$h_{max}^2 = c_o F_{max}, \tag{1}$$

where the constant c_o depends on the specific indenter shape, material constants, and loading rise time t_R ; see [Oyen 2006]. Following the discussion above, the relation (1) should be true irrespective of the maximum applied load if material points experience the same strain rates spatially affine to the maximum indentation depth h_{max} at all times. In other words, under these conditions the location of a material point with the same strain rate history scales with h_{max} . From a classical continuum mechanical viewpoint this should be fulfilled if the load F goes up from 0 to its maximum F_{max} linearly in time, as in [Oyen 2006]; in symbols, $F = (t/t_R)F_{max}$, where t_R is the load raising time. If the load is raised quadratically then $F = (t/t_R)^2 F_{max}$. If t_R remains the same while F_{max} changes, the stress fields and the strain and

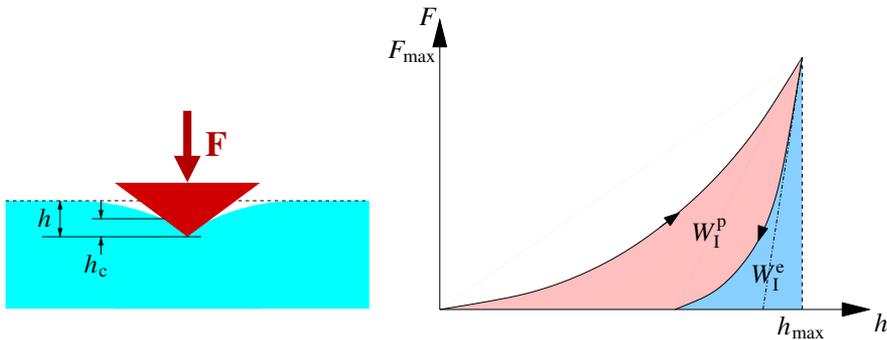


Figure 1. Indentation depths (left) and elastic W_I^e and inelastic W_I^p parts of the total indentation work W_I in a load-displacement diagram (right).

strain rate fields will be affine with respect to h_{\max} . As a direct consequence of (1) the universal hardness $H_{\max} = F_{\max}/(c_B h_{\max}^2)$ is not dependent on F_{\max} or on h_{\max} , which is the expected result from a classical continuum mechanical viewpoint in terms of local stresses and strains and their rates.

In order to examine the rate dependence of the silicone rubber various loading times were applied for a given maximum load F_{\max} . The load rising and falling time of the load-controlled indentations were chosen to be equal and set to be $t_R = 1, 10, 100,$ and 1000 seconds with zero holding time to minimize creep type deformation. The load increased quadratically in time till the maximum prescribed load was reached at t_R . The unloading was correspondingly decreased quadratically in the same time interval.

It has been found that the tested silicone material is highly elastic. Typical load-displacement curves with different indentation times are shown in Figure 2, left, including the load-displacement curves of the loading and unloading sequences. The lines in loading and unloading for these loading times can hardly be distinguished. Particularly for the 1000 second test, the indentation depth h at zero load was also practically zero, which indicates that there is little if any plastic deformation. However, to visualize that there is some dissipation, the load displacement curves in loading and unloading for the 1 second test have been redrawn in Figure 2, right.

Essentially no permanent indentation h was observed after the load had been removed, although at small loading times t_R (see Figure 2, right) some indentation depth h is measured at zero applied load. Because there is hardly any remaining indentation depth for the indentations for $t_R = 1000$ s it can arguably be assumed that most of the remaining h in Figure 2, right, at the end of the unloading sequence is actually recovered in time. This motivates the use of

$$W_I = W_I^e + W_I^v \tag{2}$$

as the decomposition of the indentation work, where W_I^v is the viscous part of the indentation work which, is not recovered.

In Figure 3 the ratio between W_I^e and W_I , that is, $\eta^e = W_I^e / W_I$, is illustrated for different indentation depths and loading/unloading times. As depicted therein, with decreasing indentation time the ratio η^e

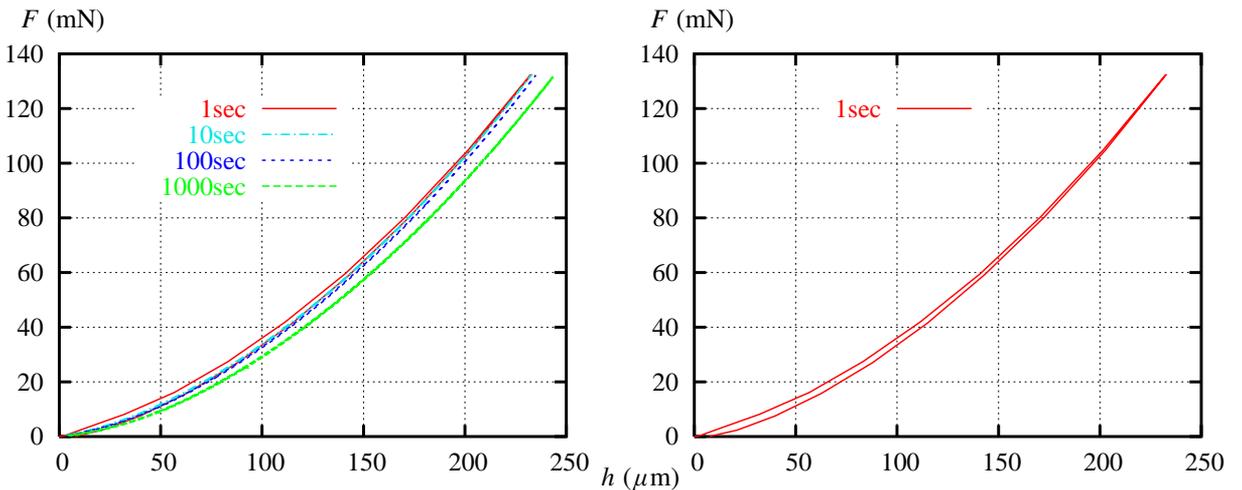


Figure 2. Indentation depth h versus load F at various loading/unloading times (left). The curves for 1 second are repeated on the right.

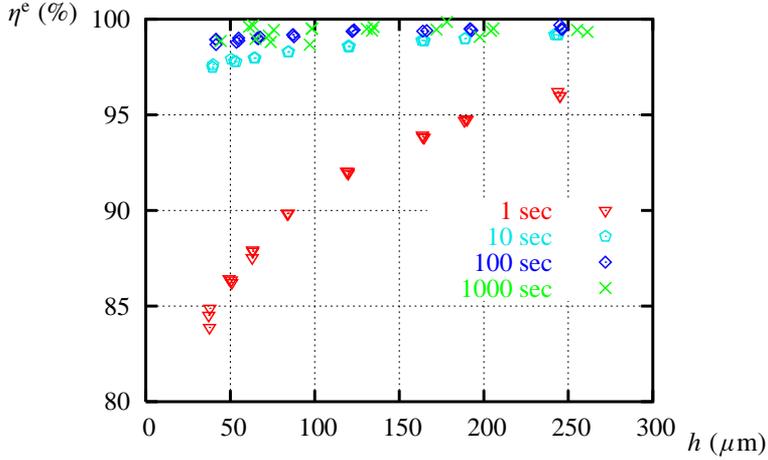


Figure 3. Ratio η^e versus indentation depth h for various loading times.

decreases, corresponding to increasing dissipation. Such behavior is well known and widely observed in other material tests such as simple tensile tests [Lemaitre and Chaboche 1990]. The values in Figure 3, however, also show a decrease of η^e with decreasing h , and therefore the dissipation of the indentation work increases with decreasing h . Similar tendencies have also been observed in other polymers [Briscoe et al. 1998; Tatiraju et al. 2008]. The magnitude with which η^e decreases is more pronounced at shorter loading times. As the majority of the deformation is elastic and hardly any plastic deformation is present the hardness values characterize elastic rather than plastic deformation.

A material model that is formulated only in local stress and strain related terms would not predict any change in η^e with the indentation depth h . The changes in η^e with h are, however, apparent in Figure 3. The indentation size effect is usually assessed by the relation between hardness and indentation depth. The values of these quantities corresponding to Figure 3 are shown in Figure 4, which plots the universal hardness,

$$H = F/A_s, \tag{3}$$

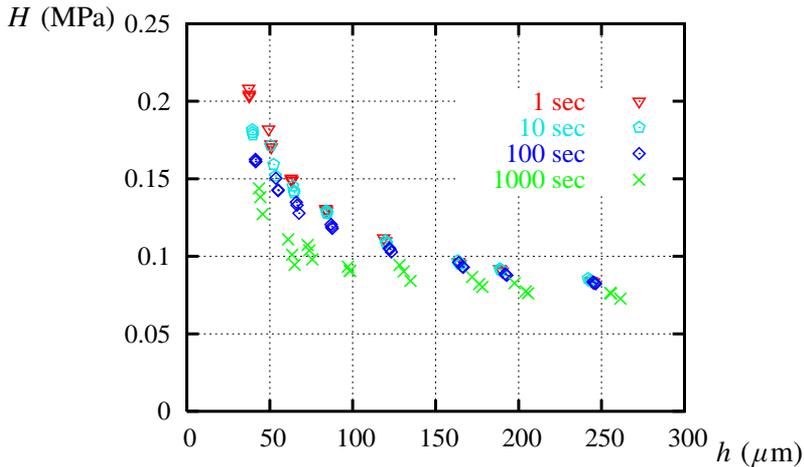


Figure 4. Universal hardness H versus indentation depth h for various loading times.

versus h for different loading times; here F denotes the force applied by the indenter and $A_s = 26.44h^2$ is the nominal contact area of the indenter penetrating beyond the zero-point of contact, h , described above [ISO 14577-1 2002].

The universal or Martens hardness was chosen because the determination of the indentation hardness has been developed for metallic materials where the elastic deformation is small compared to the inelastic deformation [Oliver and Pharr 1992], and is therefore questionable for a highly elastic material such as silicone rubber; while the universal hardness can be applied to all materials [ISO 14577-1 2002]. If size effects are not present both the universal hardness and the indentation hardness are independent of h . As can be seen in Figure 4, however, a significant increase in the universal hardness, H , has been determined for small indentation depths below about 100 microns. The figure also shows that with longer indentation times and corresponding smaller strain rates H decreases. Such increased hardness at lower loading times has also been observed by others [Shen et al. 2004], and is consistent with the increased dissipation at lower loading times shown in Figure 3. Assuming that the deformation in the considered silicone rubber is of viscoelastic nature the data in Figure 3 would indicate that the viscosity and rate dependence are increasing with decreasing h .

4. Analysis and evaluation of experimental data

In the above described experiments we observed significant indentation size effects at indentation depths between 30 and 300 microns and, with decreasing loading times, the dissipation and the indentation size effect increased. While the dissipation increases with decreasing h the elastic indentation work W_I^e is large compared to the dissipation W_I^v in all indentations performed here. For an analysis of the h -dependent universal hardness, the rate-independent hardness model suggested in [Han and Nikolov 2007] may therefore be considered as an approximation, where rate effects have been neglected.

It is well established that the deformation of liquid crystal polymers can be described by the Frank energy and the related rotational gradients [de Gennes and Prost 1993; Stewart 2004] which originate from molecular interactions. While the here-considered filled silicone rubber is not a liquid crystal polymer it is here assumed that for glassy polymers the molecular interactions related to the Frank energy still play an important role in the deformation process at small scales because the lack of nematic order is due to topological defects like cross-links and entanglements rather than to the absence of sufficiently strong molecular interactions. Following this assumption, that the Frank energy is also relevant in amorphous polymers, a Frank elasticity type rotation gradient energy was suggested by Nikolov et al. [2007] to account for the observed elastic size effects in the bending of epoxy microbeams [Lam et al. 2003; McFarland and Colton 2005]. A corresponding extension for elastoplastic materials and a related hardness model were deduced in [Han and Nikolov 2007] by including a Frank energy related indentation work term W_I^F in the total indentation work. For viscoelastic materials this extension of (2) can be given by $W_I = W_{I_\varepsilon}^e + W_{I_\varepsilon}^v + W_I^F$, where the subscript ε has been added to indicate the local nature of these indentation work components. The Frank energy density in its one-parameter form $W^F = \frac{1}{2} K \langle \nabla \mathbf{n} : \nabla \mathbf{n} \rangle = \frac{1}{2} K \langle n_{i,j} n_{i,j} \rangle$ is a quadratic function in the gradients of the orientations of the more rigid (nematic) parts of the polymer chain \mathbf{n} (averaged over all directions, which is indicated by $\langle \rangle$) and is also directly proportional to the Frank constant K , which depends on the molecular structure and properties of the polymer [Liu and Fredrickson 1993]. The change in the Frank energy density from

the initial configuration \mathbf{n}^0 to the current configuration \mathbf{n} can then be expressed by

$$\Delta W^F = \frac{K}{2} (\langle n_{i,j} n_{i,j} \rangle - \langle n_{i,j}^0 n_{i,j}^0 \rangle). \quad (4)$$

This expression is not a practical description in deformation related terms. Assuming, firstly, a decoupling of the local work densities W_ε^c , W_ε^v , and the Frank energy and that, secondly, a Frank energy related deformation potential is zero in the undeformed state and increases with deformation, [Nikolov et al. \[2007\]](#) motivated a Frank energy type deformation potential $\Delta \tilde{W}^F$

$$\Delta \tilde{W}^F = \frac{\tilde{K}}{3} \chi_{ij}^S \chi_{ij}^S, \quad (5)$$

where $\chi_{ij}^S = \frac{1}{2} (\chi_{ij} + \chi_{jj})$ and $\chi_{ij} = \frac{1}{2} e_{inm} u_{m,jn}$ represent gradients in the rotations. The displacement field is u_m and the permutation symbol is e_{jnm} . The tilde is introduced in (5) to differentiate it from the Frank energy, (4), due to the applied assumptions and approximations. In spite of the modifications of the Frank energy, (4), the K -related constant \tilde{K} in (5) should be of the same order.

With $\Delta \tilde{W}^F$, the total deformation work density is suggested as

$$W = W_\varepsilon^c + W_\varepsilon^v + \Delta \tilde{W}^F, \quad (6)$$

where W_ε^v corresponds to the indentation work component $W_{I\varepsilon}^v$ and is described in local stress and strain related terms.

To a first order approximation the magnitude of the rotation gradient χ^S averaged over the area under the indenter increases with decreasing h as the distances are proportional to h . Consequently for large indentation depths h the nonlocal work W_I^F should become small relative to $W_{I\varepsilon}^c + W_{I\varepsilon}^v$. With decreasing h , however, the energy W_I^F to be exerted during indentation will increase relative to $W_{I\varepsilon}^c + W_{I\varepsilon}^v$ resulting in higher applied forces and corresponding higher hardness values (see [\[Han and Nikolov 2007\]](#) for more details).

Based on the additional indentation work W_I^F associated with the deformation work density $\Delta \tilde{W}^F$, an approximate hardness model,

$$H = H_0 \left(1 + \frac{c_\ell}{h} \right), \quad (7)$$

was deduced in [\[Han and Nikolov 2007\]](#). It involves a length scale parameter c_ℓ and a macroscopic hardness H_0 , which also represents the lower limit of H . This model was developed by neglecting rate effects and assuming that the essential characteristics will not change with the loading time. A more general model discussed in the same paper is $H = H_0(1 + (c_\ell/h)^\gamma)$, with a fitting parameter accounting for the assumptions applied in its deduction. For $\gamma = 1/2$ this is identical to the model suggested in [\[Chong and Lam 1999\]](#). For a short range h the differences from using (7), that is, $\gamma = 1$, and $\gamma = 1/2$ are small, but they increase with the range of h .

[Equation \(7\)](#) describes the h - H relation fairly well for elastoplastically deforming epoxy, polycarbonate, and polystyrene for which experimental data of up to 10 microns were reported in the literature [\[Chong and Lam 1999\]](#). For small indentation depths (under 50 nm or so), effects from surface roughness, indenter tip curvature, and surface effects may be present, as discussed in the introduction. [Equation \(7\)](#) does not take into account these effects and consequently does not represent the size-dependent hardness below about 50 nm indentation depth.

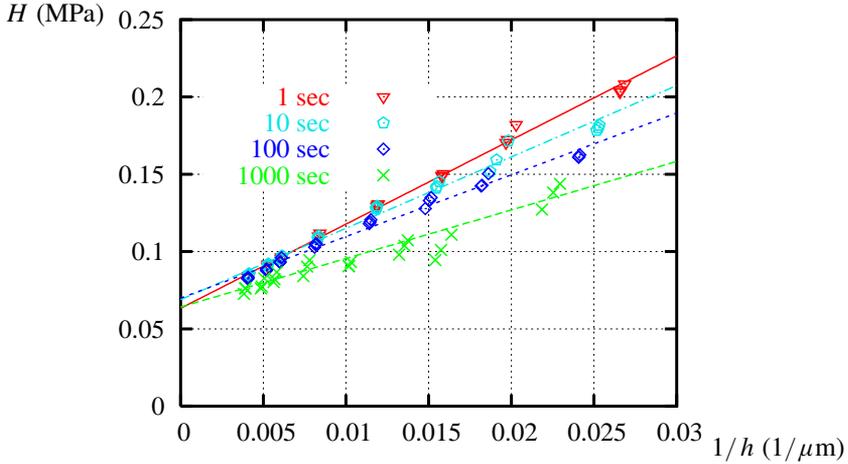


Figure 5. Universal hardness H versus inverse indentation depth $1/h$.

The dissipation is rather small in the indentation of our silicone material (see Figure 3) and therefore one could assume that the above approach should be a good approximation. In fact, (7) was also found to predict the h - H relation quite well for different loading times, as can be seen in Figure 5 where $1/h$ versus H is shown. The figure also contains linear curves in $1/h$ obtained from least square fits of the parameters of (7), that is, H_0 and c_ℓ . As can be seen from these figures the deviation of the experimental data from these linear curves in $1/h$ is rather small. As the differences in H at small loading times increase with decreasing h it can be concluded that rate effects are pronounced and thus more important at small indentations, while at large indentation depth rate effects are less important.

The numerical values of these least square fits are given in Table 1, where the macroscopic hardness H_0 varies little with the loading time, while there are quite significant increases of c_ℓ with decreasing t_R . This can also be seen in Figure 5 where the fitted lines for different t_R values more or less converge to a common H_0 at $1/h = 0$. This would imply that there is little rate dependence in the macroscopic hardness H_0 which in turn also implies that there is little rate dependence in the macroscopic deformation (that is, the deformation with or without small rotation gradients).

Loading time t_R	1 s	10 s	100 s	1000 s
H_0 (MPa)	0.0633	0.0689	0.0698	0.0633
c_ℓ (μm)	85.988	66.972	57.192	47.442

Table 1. Least square fits for H_0 and c_ℓ .

5. Discussion

Adhesion forces may be of importance at indentation depths below the micron range, as reported in [Carrillo et al. 2005] where a spherical indenter tip was used at indentation depths of about 500 nm. With a cone shaped indenter tip and in the indentation depth range considered herein, between 30 and 300 microns, the work of adhesion should, however, be less significant, or negligible compared to the

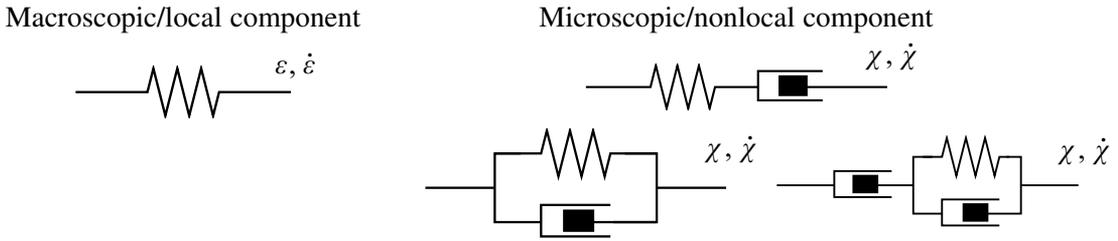


Figure 6. Rheological representation at the macro and micro levels (including the Maxwell, Kelvin–Voigt, and combined models).

indentation work components indicated in Figure 1. In this respect it is worth noting that in a numerical analysis friction effects [Mata and Alcalá 2004] could not be related to the indentation size effect. Similarly adhesion forces should also scale with the contact area and should therefore be affine with the indentation depth. These are therefore unlikely to be the cause of the indentation size effect. Using conical indenter tips the effect of the indenter tip curvature is of more relevance at submicron indentation depths [Qu et al. 2004] than the here-considered range of 30–300 microns. While rate effects were not investigated in [Lim and Chaudhri 2006] it was found that for a silicone rubber with a higher cross-link density elevated hardnesses were also observed for small indentation depths.

As opposed to [Lim and Chaudhri 2006], the silicone rubber material used in this work is not a pure polydimethylsiloxane as described in Section 2. The silicone rubber used here is more of a polymer blend, and that has not been taken into consideration in the analysis and evaluation given in Section 4. There is also a lack of clarity about the length scale of the second phases that may be present due to the fumed silica reinforced vinyl-terminated polydimethylsiloxane and the divinyltetramethyldisiloxane complex in xylene used in the fabrication process of the samples used in this work. We believe however that the issue of quantification/characterization of microscopic phases and corresponding possibly present anisotropy would be quite complicated and beyond the scope and intent of this work. Given the fact, however, that such an indentation size effect in a similar indentation depth range has also been found in [Lim and Chaudhri 2006] for highly cross-linked polydimethylsiloxane, this may indicate that the notion suggested in Section 4 is nevertheless valid for the silicone rubber used in this work as well as the highly cross-linked polydimethylsiloxane of [Lim and Chaudhri 2006]. The possible differences between the polydimethylsiloxane of [Lim and Chaudhri 2006] and the polymer blend used here fabricated with fumed silica reinforced vinyl terminated polydimethylsiloxane remains an open question which is not considered in this article.

As the depth-dependent hardness cannot be related to W_{Ie}^c and W_{Ie}^v , the small changes in H_0 at different t_R values indicate that the rate effects are mostly related to the rotational gradient-dependent W_1^F . That would necessitate a reformulation of $\Delta \tilde{W}^F$, as it should be rate-dependent and not purely elastic, as described in (5).

The viscoelastic nature of the Frank elasticity per se has already been investigated for liquid crystal polymers both in theory and experiment [Jamieson et al. 1996]. Possible combined micro-macro rheological models are suggested in Figure 6, where the macroscopic model is not rate-sensitive while the microscopic model includes damping terms dependent on $\dot{\chi}$. Note that because the macroscopic model is an elastic solid the overall local/nonlocal model would remain rheologically a (viscoelastic) solid even

if the nonlocal rheological component on its own would be a fluid. A corresponding constitutive material formulation in χ^S and its rate would necessitate a decomposition of χ^S into elastic and viscous parts. The constitutive equations and boundary conditions of such a material model would be quite complex. A phenomenological theory for elastoplastic materials with such a decomposition of a strain gradient tensor into elastic and plastic parts has been, for instance, suggested in [Chen and Wang 2002].

6. Conclusions

The rate dependence of indentation size effects in silicone rubber has been studied in the range of 30–300 microns and 1–1000 seconds. From the experimental results we can draw the following conclusions:

- The dissipation and hardness H increase with decreasing indentation depth h and with decreasing loading time.
- The rate dependence increases with decreasing indentation depth for the investigated silicone rubber. Therefore rate effects are very significant and important at small indentation depths of below 100 microns.
- The rate-independent hardness model $H = H_0 \left(1 + \frac{c_\ell}{h}\right)$ describes the hardness indentation depth relation well for a constant loading time.
- In this model the length scale parameter c_ℓ decreases with loading time, while H_0 as a projected value of the macroscopic hardness is relatively unaffected by the loading time. Consequently rate effects are mostly associated with nonlocal deformation.

Acknowledgments. The support of this work by the North Dakota EPSCoR Program (EPS-0447679) and the National Science Foundation through the CAREER program (Grant CMMI 0846692) is appreciated. The authors would also like to acknowledge the assistance of Eon-Chul Song in producing the silicone samples and the valuable comments and discussions with Dr. Hosup Jung.

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Received 24 Jan 2009. Revised 13 Aug 2009. Accepted 19 Aug 2009.

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