The "Dog Tail Test": A Quick and Dirty Measure of Yield Stress. Application to Polyurethane Adhesives.

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Abstract:
It is observed that, although consisting on very different formulations, the rheological properties of filled polyurethane adhesives may be rescaled onto simple master curves, and described with a small number of parameters: a yield stress, a low frequency elastic modulus and a characteristic time of flow. As a consequence, very simple and qualitative measurements of their deformations, such as the Dog Tail Test, may be used to deduce these parameters. By comparing the values obtained from Dog Tail Test measurements to well-controlled rheological measurements and to finite element computation, we show that such a simple and qualitative test may be used as a tool to measure both the yield stress and the elastic modulus of highly viscoelastic systems.

Zusammenfassung:
Die gezielte Steuerung der rheologischen Eigenschaften gefüllter Polyurethanklebstoffe erweist sich als entscheidend für eine qualitativ hochwertige Applikation. Trotz sehr unterschiedlicher Zusammensetzungen lassen sich die Fließeigenschaften dieser Stoffe auf ein grundlegendes Fließgesetz zurückführen und mittels weniger rheologischer Kenngrößen beschreiben: der Fließgrenze, dem Elastizitätsmodul und der Zeitabhängigkeit des Deformationsprozesses. Es ist von großem Interesse, ob diese Parameter aus den Verformungsmessungen einfacher, praxistauglicher Konsistenztests wie dem „Dog Tail Test" zu beziehen sind. Basierend auf den gemessenen, absoluten rheologischen Kenngrößen von Klebstoffen konnte das im „Dog Tail Test" beobachtete Verformungsregime unter Bemühung der Methode finiter Elemente erfolgreich abgebildet werden. Im Umkehrschluss erweist sich ein simpler Konsistenztest als tauglich, Fließgrenze und Elastizitätsmodul dieser viskoelastischen Materialien direkt abzuleiten. Der bisher nur qualitativen Aussage eines einfachen Konsistenztests kann im Ergebnis der Untersuchungen eine quantitative Komponente hinzugefügt werden. Die simplen Steifigkeitsklassifizierungen des Dog Tail Test erweisen sich als ausreichend, erforderliche Fließeigenschaften von Klebstoffen qualitativ und quantitativ zu beschreiben.

Résumé:
Bien que de formulations très différentes, les adhésifs chargés à base de polyuréthane présentent des propriétés rhéologiques qui suivent des lois maîtresses, et peuvent être décrites en utilisant un très petit nombre de paramètres : une contrainte seuil, un module élastique à basse fréquence et un temps caractéristique d'écoulement. En conséquence, on peut utiliser des expériences très simples et qualitatives de leur déformation, telles que le test de la queue, pour mesurer ces paramètres. En comparant les valeurs obtenues à l'aide du test de la queue à des mesures de rhéologie proprement contrôlées et à des simulations par éléments finis, nous montrons ainsi qu'un test aussi simple et qualitatif permet de mesurer à la fois la contrainte seuil, le module élastique de systèmes de fortement viscoélastiques.

Key words: yield stress, adhesive, filled polyurethane, finite element, modulus, creep, viscosity

1 INTRODUCTION

Industrial laboratories are notorious for using a range of tests often described as qualitative and of which the sole merit is often thought to lie in comparative studies. Those tests are not directly correlated to true or quantitative physical measurements but are "quick and dirty" tests that are simple, fast and cheap to carry out. Comprehension of these tests is however needed in order to optimize their use or avoid misinterpretation.

One such test is used daily around the world on probably every single construction site using concrete. It is the renowned Slump test which consists in measuring the height drop of a conical mould previously filled with concrete after the mould is lifted. For many years this test has

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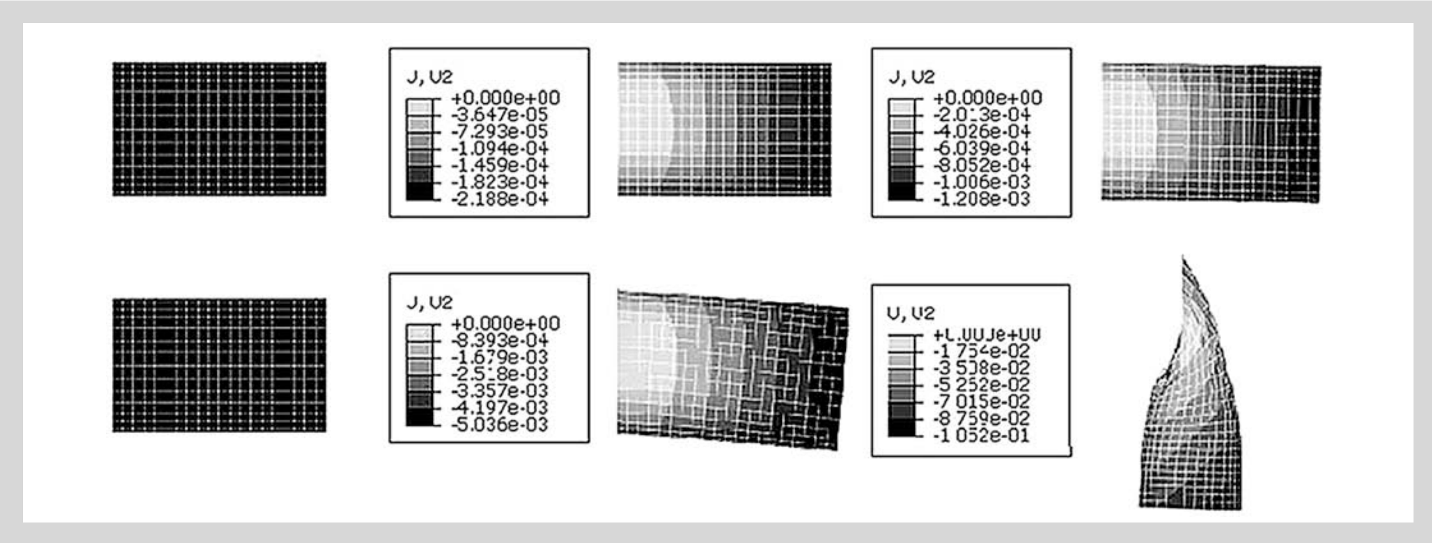

Figure 9: Displacements (in m) in z direction along the Dog Tail Test. From the left to the right: no deformation, elastic deformation, creep deformation. Upper cylinder at t = 150 s, product D5. Lower graphics at t = 150 s, product D1.

$$\overline{\dot{\epsilon}_{\sigma}} = A\bar{q}^{n}t^{m} \tag{1}$$

Where *A*, *n* and *m* are parameters to identify with experiments. For a maximum accuracy, data must cover the time domain of interest in the analysis which is about 100s for the Dog Tail Test. The the stress applied in the test must be close enough to the stress applied in the real application (between 0 and 589 Pa here). Integrating the time hardening creep law over time at constant stress brings:

$$ln(\overline{\epsilon_{\sigma}}) = \left[ln\left(\frac{A}{m+1}\right) + n\,ln\left(\bar{q}\right)\right] + \left(m+1\right)ln\left(t\right) \tag{2}$$

Identification of the three parameters *A*, *n* and *m* is carried out with rheological creep tests represented in Figure 8. Two series of creep measurements are linearly fitted at 400Pa and 700Pa. Finally, we measured for the suspension D5: $A = 9\cdot10^{-9}$, $n = 1.47$, $m = -0.58$, and for the suspension D1: $A = 3.2\cdot10^{-8}$, $n = 2.1$, $m = -0.61$.

The evolution of the 3D shapes of the two types of samples is represented in Figure 9 as a function of time. Figure 9 represents from the left to the right (i) the undeformed states, (ii) the states after elastic loading and (iii) the states after the creep response in both cases marked 1 and 5. The gray scale represents the evolution of the vertical displacement of the material (from dark to light). The general shape of the experimental Dog Tails is thus recovered, and marks ranging from 1 to 5 may be simulated, when one takes into account both the elastic and the flow constitutive equations of the system. These simulations show that, in a time of 2 minutes the Dog Tail Test quotation, ranging from 1 to 5, corresponds to elastic moduli ranging from 10^6 to 10^4 Pa.

Nose test mark	Yield stress (Pa)	Plateau elastic modulus (Pa)	Characteristic time (s)
1	> 200	$> 2\cdot10^5$	> 100
2	90	$1.5\cdot10^5$	100
3	50	7000	30
4	30	1000	10
5	< 20	< 800	< 10

Table 1: Characteristic yield stresses, elastic moduli at low frequency and flow times of the studied polyurethane adhesives, corresponding to nose test marks ranging from 1 to 5.

4 CONCLUSIONS

The "Dog tail" test assesses the "stiffness" of a product by ranking with discrete values in a scale of 1 (very good) to 5 (very bad). We have seen that, for all tested materials; this result correlates well with an apparent yield stress. The possibility of superposing of the flow curves makes it possible to determine a characteristic time related to the deformation of the network formed by the solid particle. This time is found to be well related to the results of the pragmatic Dog Tail Test. Finite element simulations have been used to simulate the evolution of the shape of the Dog Tail as a function of the rheological properties of the system. It has been shown that not only the elastic properties but also the flow properties measured with rheological creep test of the adhesives should be taken into account to describe the complete rheological behaviour of the adhesives. Results obtained from the Dog Tail Test correlate well with characteristic times deduced from creep measurements. We have thus shown that it exists a relationship between the nose test mark on one hand and the yield stress, the elastic low frequency modulus and the characteristic flow time of our polyurethane adhesives, on the other hand. This relationship is summarized in Table 1. Moreover, the Dog Tail Test is much faster to perform, and thus remains a useful quick and dirty method of assessing the rheological properties of adhesives.

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