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High strain rate testing at the nano-scale: A proposed methodology for impact nanoindentation

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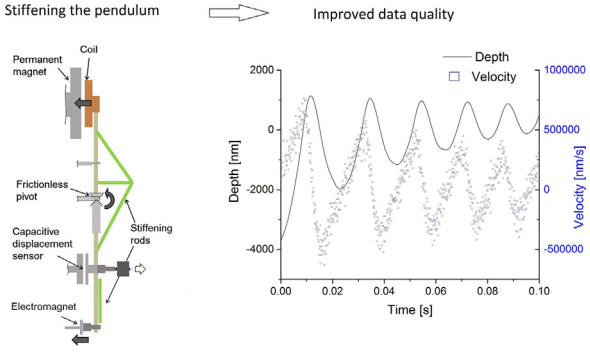
HIGHLIGHTS

- Impact indentation measures dynamic hardness at strain rates up to 10^3 s^{-1}.
- We outline its applicability and determination for hard, brittle materials.
- We discuss the accuracy with regards to surface determination and strain rate.

GRAPHICAL ABSTRACT



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ABSTRACT

Hard, brittle materials are often subjected to mechanical loading on the nano-scale at high strain rates, and while high loading rates can be achieved on the macroscopic scale, there are few methods in the micro-mechanical regime in which these materials can be plastically deformed.

Impact nanoindentation is a possible method which retains the flexibility of quasi-static nanoindentation, namely that it is useable for a wide range of materials and can be used to test small phases in a site-specific manner while still applying high strain rates. It therefore helps elucidate deformation mechanisms in regimes that were not accessible up to now. However, previous investigations suffered from limitations regarding the data acquisition, subsequently reducing the scope and accuracy of the determined properties.

Here, an improved experimental setup is used and a systematic analysis is described which analyses the energy loss, indentation depth recovery and dynamic hardness over a wide range of strain rates. The accuracy of the results is investigated by atomic force and optical microscopy, compared to the accuracy of other approaches and discussed.

1. Introduction

In normal (quasi-static) nanoindentation the accessible strain rates are quite limited, with most measurements falling in the range of 0.1–0.001 s^{-1} [1]. This does not reflect the loading conditions materials have to face in reality, with brittle materials often failing during loading at high strain rates, e.g. falling to the ground or being hit by small objects with high velocity. Standard high-velocity testing methods, e.g. Charpy-impact testing, use macroscopic sample dimensions [2], but for brittle materials like glasses, it is well known that failure is controlled by pre-existing defects, such as small cracks and notches, which commonly depend on the manufacturing process and reduce the critical load for failure to drastically below the intrinsic material strength [3–8]. The latter

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may then only be estimated where cracking is suppressed and plasticity activated instead. This normally occurs at high temperature (where plastic deformation becomes easier) or under confining pressure (where cracking is rendered less favourable). At high temperatures, fibre pulling or conventional macroscopic compression experiments can be used and applied up to high strain rates [9–15]. At low temperatures, tests such as uniaxial testing result in fracture and a confining pressure must be introduced to suppress it. This can either be achieved in triaxial experiment using a confining medium or, much more simply, in using indentation or a similar geometry where the surrounding material provides the confinement. Where the scale of the test is additionally reduced, the measured results become less representative of pre-existing flaws and instead dominated by the intrinsic material properties. There are small scale impact testing methods which allow testing (and in application also machining or processing) of brittle materials at high rates and at room temperature, e.g. shooting small, hard spheres at glass surfaces, such as in shot-peening [16–19]. But even by the use of high speed cameras, the process of plastic deformation induced during impact is hardly observable, leaving many questions open.

In order to combine the advantages of a small-scale impact with a quantitative measurement, impact nanoindentation testing can be used. It significantly expands the range of accessible strain rates compared with quasi-static nanoindentation and retains the small scale and confining pressure that inhibit cracking and hence permit the characterisation of plastic deformation. There are several publications using a pendulum-based nanoindentation system (from MicroMaterials Ltd.) to perform impact nano-indentation experiments [20–33], as was also done in this work. Recently, Phani and Oliver also demonstrated high strain rate nanoindentation experiments [34] performed using a "traditional" nanoindentation setup, whereby a step load applies the desired force to the sample in ~0.5 ms after a surface-find step, i.e. without impact. Despite the difference in setup, the data obtained at high strain rates nevertheless also shows oscillatory behaviour due to the dynamic nature of the test. It is therefore hoped that some of the analyses presented herein are useful for general high strain rate testing.

It should be noted that although broadly similar techniques - the indentation of a surface using a sharp tip - there are significant differences between quasi-static and impact nanoindentation. Quasi-static nanoindentation continually measures - and can continually control - indentation load and displacement, with all the subsequent analysis parameters (typically hardness and modulus) derived from the depth-load curve. On the other hand, impact indentation uses the depth-*time* curve, such that the key parameters are now *acceleration* and displacement. From these parameters, velocity and therefore kinetic energy (and its loss) can be determined. There is also a difference in the applied load in both techniques. In impact indentation, the set load is simply one factor (along with the accelerating distance) that controls the impact energy, and is not connected to the load in quasi-static indentation. Additionally, this load is continually applied throughout the test, such that the measured depths are under load (discussed later in Fig. 4).

There are two types of investigation which may be envisaged with reference to deformation or failure of brittle materials in application and during processing: failure during a single, catastrophic impact event and damage accumulation during repetitive impact at or near the same site. Both can be studied by impact nanoindentation and conducted in two ways (assuming here the use of a pendulum-based system): i) using a high data acquisition rate of several kHz, the changes in velocity and displacement are measured and interpreted as changes in energy. A dynamic hardness may then be determined and investigated for a single acceleration of the tip into the sample in great detail; ii) employing a lower data acquisition rate but multiple (up to hundreds of) impacts in a row where the tip is accelerated before every new impact. The changes of impact depth and any critical depths or rates of growth of the impact damage site are measured. Here, these methods are referred to as 'dynamic hardness' and 'multiple impulse' methods, respectively.

When using the dynamic hardness approach, the tip is only retracted once. After reaching the sample, the tip will penetrate the sample and the kinetic energy is then transferred into elastic and plastic deformation. Once all the energy is transferred, the elastic deformation will be reversed, resulting in a springback of the tip. The springback height will depend on the ratio between plastic and elastic deformation. Using the multiple impulse setup, the tip is retracted and accelerated repeatedly, resulting in repetitive impact indentations with the same kinetic energy. This way, the maximum indentation depth over a number of indents can be analysed, allowing the onset of cracking, delamination or other failure mechanisms in a coating or bulk material to be studied. The multiple impulse method can be used to investigate the cracking behaviour of hard materials under repetitive loading and has been used in numerous publications [20–28,30,32,33].

The dynamic hardness method, on the other hand, offers superior, detailed information that can be used to investigate the damping behaviour, the amount of elastic and plastic deformation and the dynamic hardness of a material. However, until now there is very little work published using this latter method [28,31,33,35]. The reason for this is that the data analysis requires accurate time-resolved data, but oscillations developing in the system during or after the first impact have been found to be so strong that they make it almost impossible to obtain accurate position measurements [33]. There are a few publications that use this method to investigate the mechanical response of soft materials like 1100 aluminium [35] or fine-grained, solid-solution-hardened magnesium [31]. What these publications have in common, however, is that they deal with soft materials and low loads and identify several sources of (potential) inaccuracies [31], which require as part of the analysis the assumption that only the first impact introduces plastic deformation [33] or the need to use extensive models to fit the velocity data [35]. However, Jennett et al. showed that by applying stiffening rods to their pendulum-based system, the oscillations could be significantly diminished even if hard materials are used at high loads [28]. This way the velocity-depth data are accurate enough so that they can be used to determine parameters like kinetic energies and dynamic hardness without fitting curves – and therefore assumptions – to the data, as we recently have shown in a variety of glassy materials [36]. This modification is of special interest as the dynamic hardness method is typically used to study the impact deformation of coatings that exhibit a very high hardness [20–22,24–27,30,32,33]. Unless an analysis method is applicable to these challenging materials, it will be difficult to use efficiently and universally.

Up to now, there is no standard procedure for how to perform and analyse impact nanoindentation experiments for a variety of materials, especially for key analysis steps: the determination of the surface, dynamic hardness and representative strain rate. In this paper, therefore, we map out possible parameters for analysis and how these are derived from the raw data. An extended analysis of the data recorded in nanoscale instrumented indentation tests is presented, which includes the in-depth analysis of not only the final material response after several rebounds and re-impacts but also the response of the material during the first impact – as more commonly relevant in impact loading, where subsequent impacts of the same object are not normally experienced at the same site. Using an experimental setup combined with the improvements from Jennett [28], the methods described here are applicable to all materials without the use of fitting or modelling, reducing potential inaccuracies and simplifying experimental analysis.

In addition, an exploratory study is presented using both impact methods. In order to include both changes in plastic deformation and cracking in the presented data, we have chosen a sodium-borosilicate (NBS) glass in two different microstructural states, highlighting the ability to reveal differences even in two intrinsically very similar materials in which the ability to understand and predict deformation and cracking at ambient temperatures and high strain rates is essential for materials design.