

## **Mechanical properties of aluminum alloys by instrumented indentation: case study on Almigo Hard**

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### **Introduction**

Conventional hardness measurement has been used for many years for characterization of many types of materials including metals, ceramics and certain plastics [1-3]. The most widely used methods are Brinell and Rockwell hardness using sphero-conical indenters and Vickers and Knoop hardness measurements using pyramidal indenters. While Rockwell and Brinell methods are often used at high loads (hundreds to thousands of Newtons), Vickers and Knoop methods are often used also at low loads, i.e. tens and units of Newtons. All these methods are based on optical inspection of the residual imprint and their application is therefore limited to dimensions that are visible under optical microscope. Optical inspection also hinders to some extent automation of the measurements by computers, although there has recently been progress in image analysis and automatic measurements of the residual imprints, especially in the case of Vickers microindentation.

Several decades ago, the first attempts to use other methods for measurements of hardness of materials and especially that of thin films, have been done. These methods were based on the theory of contact developed by Hertz, further extended by Johnson and Sneddon [4] and transformed into procedures applicable in the experimental praxis by Doerner, Nix, Field, Swain and Oliver and Pharr in the 1990s [5-7]. Using this method the materials properties are extracted from the analysis of the load-displacement curve obtained during penetration of loaded indenter into the material. The method, named instrumented indentation, significantly reduces the time required for hardness measurement and opens new perspectives for characterization of materials at very low scale. Widespread of instrumented indentation has been greatly facilitated by the expansion of personal computers, which allowed for convenient and fast analysis of the load-displacement data. In general, there are three main advantages of instrumented indentation in respect to conventional indentation:

- Possibility of characterization of materials at very small loads (down to several  $\mu\text{N}$ ) thanks to very precise displacement and load sensing systems.
- More materials characteristics (hardness, Young's modulus, creep, elastic/plastic work of indentation, etc),

- Automated analysis of the load-displacement data and application of various loading profiles (cyclic, depth controlled, etc).

Instrumented indentation (or shortly 'nanoindentation') became very important especially with the development of thin films and advanced materials with various micro- and nanostructures since it allows assessment of mechanical properties on a very small scale (tens or hundreds of nanometers). To obtain correct results even at such low depths, the three-sided Berkovich indenter is often used instead of the Vickers indenter because of its better compliance with the ideal geometric shape [7]. Spherical indenters are used for elastic-plastic characterization of materials and cube corner indenters are usually used for fracture toughness measurements.

This study aims to demonstrate the main advantages and features of instrumented indentation by various measurements on a special aluminum alloy Almigo Hard. The Almigo Hard alloy belongs to a new class of aluminum 7000 series with T651 temper and it is used because of its high strength along with excellent machinability, thermal conductivity, high polishability and good shape stability. Among the most important applications of this material belong moulds for injection and compression forming as well as bolsters and force plates used in high load strain gauges. The nanoindentation measurements would allow fast local characterization of hardness, Young's modulus and other important materials properties such as elastic to total energy of indentation ratio. To our knowledge, the Almigo Hard alloy has not yet been characterized by nanoindentation and this study will therefore bring a new insight into determination of local properties of this particular alloy, indispensable for proper designing of components such as strain gauges.

### **Experimental setup**

The composition of the Almigo Hard (Alcan, France) alloy is (at. %): Cu 1.9, Mg 2.2, Zn 8.4, Zr 0.1, Al remainder. The material was heat treated by T651 temper. Samples in form of cylinders of 20 mm diameter and 15 mm height were polished using standard metallographic procedures with diamond paste and electrolytically polished in 5% perchloric acid solution at 40V. Indentations were performed on this polished surface. For comparison, two reference materials were prepared and characterized using the same methods: D16CT1 Al alloy (AlCu4Mg1 type) used in aircraft construction and 15Ch2MFA ferritic steel used in nuclear reactor pressure vessels [8].

The characterization of the samples was performed by Nanoindentation Tester (NHT, CSM Instruments, Switzerland) using instrumented indentation technique with maximum loads ranging from 1 mN to 500 mN to characterize the materials on a large scale. Single load indentations were performed with the following maximum loads: 1 mN, 2 mN, 5 mN, 10 mN, 50 mN, 100 mN, 200 mN and 500 mN. Both loading and unloading times were set to 30 s with a 5 s hold at the maximum load. There were six indentations performed in several areas on each sample.

Cyclic loading-partial unloading indentations were used for comparison with the single load indentations. The parameters of each cycle in the cyclic indentation were as follows: loading time 10 s, hold period 5 s, unloading time 10 s and unloading to 20 % of the maximum load in the given cycle. There were 20 cycles in one cyclic measurement and each measurement started with load of 2 mN and ended with load of 500 mN. Diamond Berkovich indenter was used for most of the indentation measurements; spherical diamond indenter with 20  $\mu\text{m}$  radius was used to obtain the elastic-plastic response of the tested materials. Spherical indentation was performed only using cyclic indentation method. The results of indentation results obtained with Berkovich indenter were calculated according to ISO 14577 standard [9], the results of spherical indentation were obtained using Tabor's approach [9].

## Results

The results of the nanoindentation experiments showed that most of the indentations would be very difficult or nearly impossible to visualize and measure using optical methods. Figure 1 demonstrates the sizes of residual imprints after the nanoindentation experiments with the maximum load varying from 2 mN up to 500 mN. Note that it was impossible to locate the 1 mN indent in the optical microscope due to its small size. The use of nanoindentation based on analysis of the load-displacement curve for obtaining mechanical properties of the tested material was therefore indispensable.

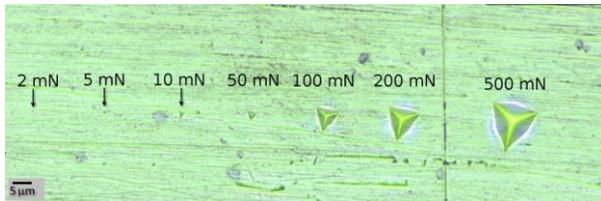


Figure 1: Optical image of a row of indents obtained with maximum loads of 2, 5, 10, 50, 100, 200 and 500 mN.

electrolytically. The results of nanoindentation on this sample showed that hardness remained constant at all indentation loads. It was therefore decided to prepare also the Al alloy and reactor steel samples by electrolytical polishing in order to avoid the effect of surface hardening.

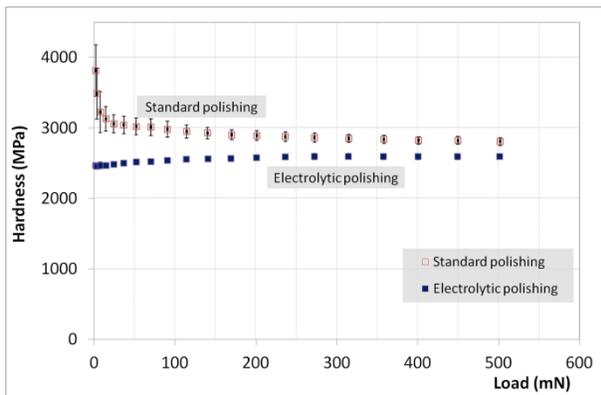


Figure 2: Hardness profile for increasing indentation load for conventionally polished sample (hollow symbols) and electrolytically polished sample (full symbols).

efficient and time-saving alternative to a large series of single load experiments. This agreement was further verified by comparison of single load indentation results with cyclic indentation results on the three tested samples. In all cases the hardness values obtained from single load indentations were in excellent agreement with hardness values obtained from cyclic loading indentations (Fig. 4). Similar agreement between single load and cyclic loading indentation results was also found for Young's modulus on all samples.

### Sample preparation: influence of surface hardening

The nanoindentation measurements on the Almigo Hard sample polished on emery paper and diamond paste (standard polishing) and electrolytically polished regions showed hardness increase at lower loads (see Fig 2). This effect is known as surface hardening due to plastic deformation that is induced in the material during polishing. To remove this hardened layer, the sample was polished

### Cyclic loading versus single load indentation

Instrumented indentation allows applying various loading profiles, which can be advantageously used for reduction of the time needed for testing. However, it has to be ensured that the cyclic method yields the same results as the quasi-static method using only single maximum load. The graphs in Fig. 3 show comparison of typical load-displacement curve for a 500 mN single load indentation and cyclic indentation with 500 mN maximum load on the Almigo Hard, Al alloy and reactor steel samples. Note excellent agreement of both single load and cyclic curves for all three samples. These results confirm the possibility of using the cyclic method as an

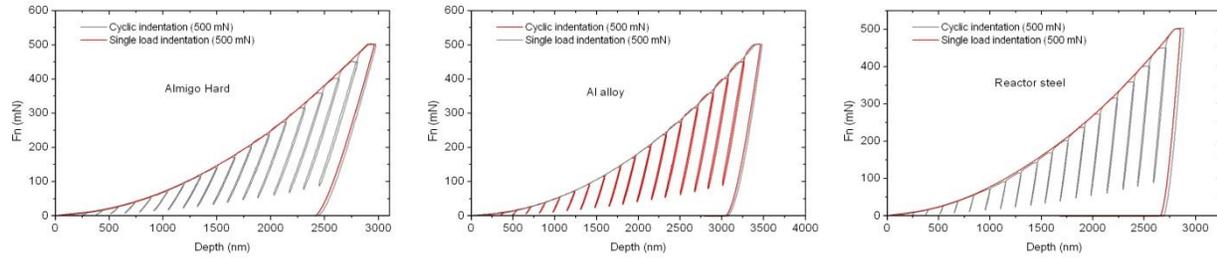


Figure 3: Comparison of cyclic indentation method from 2 mN to 500 mN and single load indentation to 500 mN: Almigo Hard (left), Al alloy (centre) and reactor steel (right).

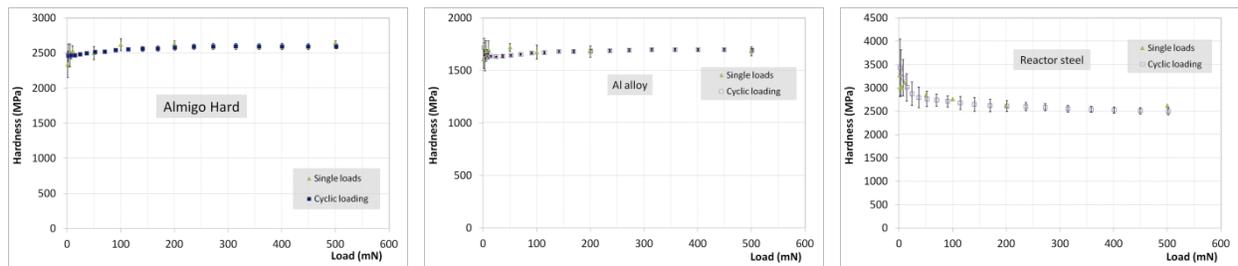


Figure 4: Comparison of hardness values obtained by cyclic indentations and hardness values obtained by single load indentations: Almigo Hard (left), Al alloy (centre) and reactor steel (right).

### Elastic-plastic characteristics obtained by spherical indentation

Cyclic indentation method with spherical indenter with 20  $\mu\text{m}$  radius was used for determination of elastic-plastic behaviour of the three tested materials to obtain quickly a sufficient number of data to plot the corresponding representative stress-strain characteristics. In the first approximation, data from spherical indentation were analyzed using Tabor's equations that relate the average stress and contact radius under spherical indentation to representative stress and representative strain [10]:

Representative stress:  $\sigma_{repr} = \frac{P}{C \pi a^2}$  where  $P$  is the load,  $a$  is the indentation radius and  $C$  is the constraint factor ( $\sim 3$  for most engineering materials).

Representative strain:  $\varepsilon_{repr} = 0.2 \frac{a}{R}$  where  $R$  is the radius of the spherical indenter; pre-factor 0.2 was determined empirically in [10]. The results are shown in Fig. 5 where the so-obtained representative stress-strain characteristics are plotted. These outcomes were completed by calculation of the elastic to total work of indentation ratio from Berkovich indentations (Fig. 6), which indicates the level of elastic recovery during indentation.

### Discussion

Due to the size of the indents (see Fig. 1), conventional microhardness was not suitable for local characterization of the tested materials and instrumented hardness had to be used. The instrumented indentation showed that it is much more versatile method which reveals also other materials properties than hardness. Using spherical indenters, the elastic-plastic characteristics of the tested material can be estimated using instrumented indentation. However, since low loads and low indentation depths are often used in instrumented indentation, care has to be taken about the surface preparation. Namely for metallic samples, metallographic polishing often leads to undesired hardening of the near-surface

regions. As compared in Fig. 2, hardness obtained on the same sample prepared by standard polishing and electrolytical polishing shows considerable differences, especially in the low load/low depth range. The samples for nanoindentation therefore have to be properly prepared otherwise the results might reflect the influence of the preparation method rather than properties of the material.

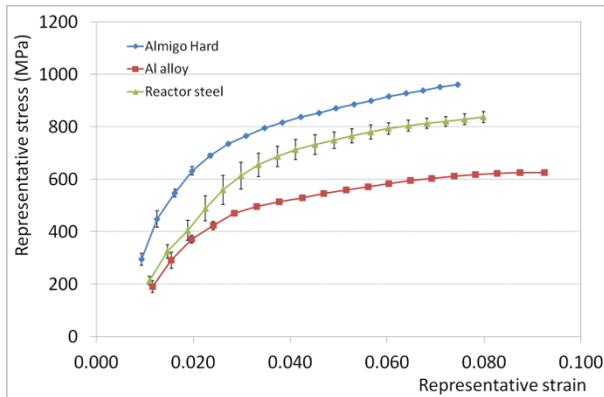


Figure 5: Comparison of representative stress-strain characteristics for all three tested materials.

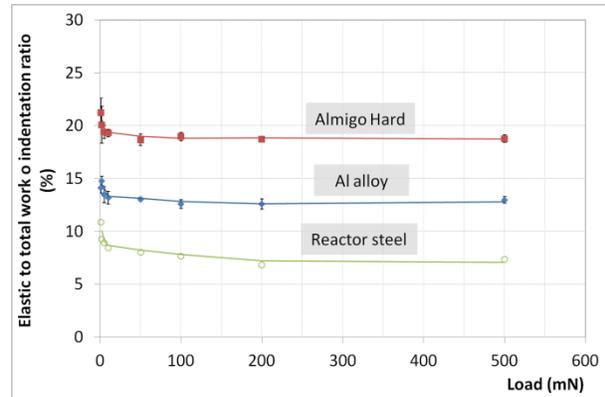


Figure 6: Comparison of elastic to total work of indentation ratio for all three tested materials.

One part of this study was dedicated to confirm whether the cyclic loading/unloading indentation can be used as an efficient and time saving alternative to large number of single indentations at different loads. Several series of cyclic and single load measurements on all three samples proved that cyclic method yields almost the same results as single load indentations (see Fig. 3 and Fig. 4). This excellent agreement was found despite different loading rates in the cyclic and single load methods: the loading and unloading times for single load indentation were both set to 30 s while the loading and unloading times in each cycle were set to 10 s (the hold period of 5 s in each cycle was maintained for both single load and cyclic indentation). It can be supposed that cyclic indentation yields the same results as single load indentations for metallic and other materials not exhibiting significant time dependent mechanical properties.

For both electrolytically polished aluminium alloys the hardness remained constant with depth whereas for reactor steel (Fig. 4 right) the hardness was increasing with decreasing depth. This increase in hardness is very likely due to the indentation size effect (ISE) which leads to apparent increase in hardness at low loads (see Ref. [11]). Young's modulus, on the other hand, remained constant independently of normal load for all three tested samples.

Single load indentations revealed also the elastic to total work of indentation ratio which characterizes the ability of the material to recover elastically after the indentation (Fig. 6). Out of the three tested materials, Almigo Hard exhibited the highest degree of this elastic recovery (19 %) while Al alloy showed ~13 % of elastic recovery and reactor steel only ~7 % of elastic recovery (see Fig. 7). The high elastic recovery of Almigo Hard is due to specific composition and heat treatment of this alloy and it confirms the utility of this alloy for advanced applications as an alternative to steel.

Spherical indentation was used for estimation of elastic-plastic behaviour of materials. Although it cannot be used as a substitute to standard tensile testing because the stress-strain field is different during spherical indentation (compressive stresses), it allows estimation of yield strength especially for small samples. It also allows local characterization of elastic-plastic behaviour of single grains which would otherwise be very difficult or impossible to obtain by standard tensile testing.

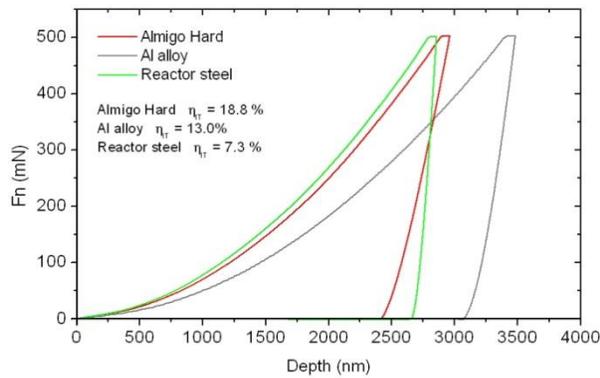


Figure 7: Comparison of elastic to total work of indentation ratio and indentation load-displacement curves for 500 mN single load indentations.

## Conclusions

This paper presents a case study of instrumented indentation for assessment of mechanical properties of Almigo Hard aluminium alloy, Al alloy and ferritic reactor steel. It shows clearly that conventional microhardness is not suitable for small load measurements and that the instrumented indentation can reveal significantly more information about the tested material such as Young's modulus and elastic-plastic characteristics on a large scale. The study allowed comparison of mechanical properties of the three tested materials in order to demonstrate the superior properties of the Almigo Hard alloy for advanced applications such as moulding and high load strain gauges.

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