

Analysis of the Application of the Instrumented Indentation Test for Aluminum and Its Alloys

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Abstract

This paper considers the specific features of instrumented indentation test as applied to aluminum and aluminum alloys. Particular attention is paid to the pile-up/sink-in effects, strain hardening, surface roughness, thickness of the investigated layer, and indenter geometry. Published results on pure aluminum, aluminum alloys, aluminum coatings and joints, as well as studies aimed at improving the processing of indentation load–displacement curves $P(h)$, are analyzed. The paper also presents an experimental study of AMg2 aluminum alloy (AA5052). At the load of 20 mN, the mean indentation hardness and indentation elastic modulus for the polished surface were approximately 0.959 and 75.8 GPa, respectively, whereas for the unpolished surface they were 0.865 and 72.5 GPa. The most pronounced effect of roughness was not so much a systematic shift in the mean value as an increase in the scatter of the results, particularly for hardness. The experimental results are compared with published data and discussed in terms of the applicability of instrumented indentation to the characterization of aluminum and its alloys.

Keywords: Instrumented indentation test; Aluminum alloys; Hardness; Elastic modulus

1. INTRODUCTION

Aluminum and its alloys are among the most widely used metallic materials in modern industry because of their combination of low density, corrosion resistance, manufacturability, and the ability to achieve a wide range of mechanical properties [1]. At the same time, the service performance of aluminum materials depends not only on the nominal alloy grade but also on the material condition, heat and mechanical treatment, grain size, the presence of secondary phases, and the condition of the surface layer.

The service reliability of metallic materials is traditionally assessed using tensile, hardness, impact-toughness, and other standardized tests. However, such tests require relatively large material volumes and generally characterize properties averaged over the specimen. Instrumented indentation is a promising method for local mechanical characterization because it records the relationship between applied load and indenter displacement and allows hardness and elastic properties to be calculated

from the resulting curve [2]. The relationship between mechanical properties and the structural and deformation state of a material is nevertheless essential for interpreting local test results. For example, studies of structural materials have shown that the mechanical state produced by prior processing and hardening directly affects the local resistance of a material to plastic deformation [3]. Consequently, indentation testing of aluminum alloys requires their pronounced sensitivity to strain hardening to be considered. Another important source of data scatter is the indenter itself. Even Berkovich indenters with the same nominal geometry may differ in their actual tip shape and tip radius. The authors of Ref. [4] showed that different Berkovich indenters can produce systematically different hardness values. This is particularly important at shallow indentation depths, where the actual tip geometry has a significant effect on the contact area. The need to account for experimental-method features in local mechanical characterization was also demonstrated in Ref. [5]. In general, the load–displacement curve, i.e., the indentation

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curve $P(h)$, contains information not only about the material properties but also about contact geometry and the characteristics of the measurement system. One of the key elements of the procedure is determination of the indenter area function. The authors of Ref. [6] proposed direct determination of the indenter-tip geometry using optical surface-topography measurement instruments, thereby reducing the uncertainty associated with indirect calibration. A similar approach was developed for atomic force microscopy in Ref. [7], demonstrating the potential for high-accuracy calibration of indenter geometry.

Another research direction involves mathematical reconstruction of indenter geometry. Study [8] demonstrated the use of interpretable machine-learning models to predict the radius of a nanoindenter tip. Automated processing of indentation curves is also being developed. In Ref. [9], a genetic algorithm was applied to load–displacement data, demonstrating the potential for automating the identification of mechanical properties. Aluminum is particularly important in applications where the properties of local regions or surface layers, rather than those of bulk material, must be assessed. For example, an AZ31B/AA6061 joint was investigated in Ref. [10], where local mechanical characterization was used to analyze the welded-joint structure. The mechanical and tribological properties of a modified Al-1100 surface were studied in Ref. [11], demonstrating the practical relevance of local characterization of aluminum surface layers. Aluminum materials are also used in more complex composite systems [12,13], where composite systems containing aluminum and intermetallic or magnetic constituents were investigated, highlighting the need to relate local mechanical properties to microstructure. For deposited aluminum layers, instrumented indentation makes it possible to assess properties directly within the coating. For example, study [14] investigated AA1100 produced on a substrate by spot friction surfacing. In Ref. [15], indentation was used to investigate nanocrystalline Al–V alloys produced by high-energy ball milling followed by spark-plasma sintering. The authors related microstructure to hardness and elastic modulus: nanocrystalline Al-2V, Al-5V, and Al-10V were examined after processing at 200...400 °C. For example, nanoindentation of Al-5V at 200 °C yielded $H = 5.21 \pm 0.36$ GPa and $E = 96.21 \pm 2.47$ GPa, while Al-10V yielded $H = 6.24 \pm 0.23$ GPa and $E = 108.53 \pm 3.62$ GPa. Hardness decreased with increasing processing temperature. Thus, instrumented indentation is particularly useful for materials whose mechanical properties vary substantially on a local scale.

Recent studies of AA6061 show that even the unloading portion of an indentation curve may depend on grain structure and the scale of deformation heterogeneity [16]. For AA5083, nanohardness was investigated within a gradient plastic layer produced by localized high-pressure

processing, directly demonstrating the applicability of nanoindentation to spatially resolved property measurements [17]. Indentation creep is also important for aluminum alloys. In Ref. [18], the creep response of AA6060 was studied as a function of aging conditions and processing temperature. At the same time, improved nanoindentation procedures based on continuous strain-rate variation have been proposed for investigating strain-rate sensitivity [19], including time-dependent creep behavior [20].

Thus, modern instrumented indentation of aluminum is not merely a method for determining two parameters—hardness and elastic modulus. The results depend on indenter geometry, material structure, plastic-deformation behavior, loading rate, hold time, surface roughness, and the thickness of the tested layer. The aim of this work is to systematize these factors, with particular emphasis on surface roughness and the deformation behavior of aluminum, and to experimentally assess the effect of roughness on the determination of hardness and elastic modulus.

2. INSTRUMENTED INDENTATION TEST

2.1. General principles of instrumented indentation

Instrumented indentation is based on recording the load applied to an indenter and the corresponding displacement of its tip. Unlike conventional hardness tests, in which the final residual impression is analyzed, instrumented indentation uses the complete load–displacement curve $P(h)$, including both loading and unloading. During loading, the indenter penetrates the material and initially produces predominantly elastic deformation followed by elastic–plastic deformation. After the maximum load is reached, unloading is performed. The shape of the unloading branch depends on the elastic properties of the material and the contact geometry. The classical and most widely used procedure for processing indentation curves was developed by Oliver and Pharr [21]. Their original work proposed a method for determining hardness and elastic modulus (Young's modulus) from load and indenter–displacement data. Subsequent development of the method refined the physical interpretation of contact depth and the indenter area function [22]. Indentation hardness is calculated as the ratio of maximum load to contact area:

$$H_{IT} = P_{max} / A_p, \quad (1)$$

where H_{IT} is the indentation hardness, P_{max} is the maximum load, and A_p is the projected contact area at maximum load. A key parameter is the contact depth h_c , which does not necessarily coincide with the maximum measured depth h_{max} . In the Oliver-Pharr method, it is determined from the maximum depth, load, and contact stiffness:

$$h_c = h_{max} - \varepsilon \times (P_{max} / S), \quad (2)$$

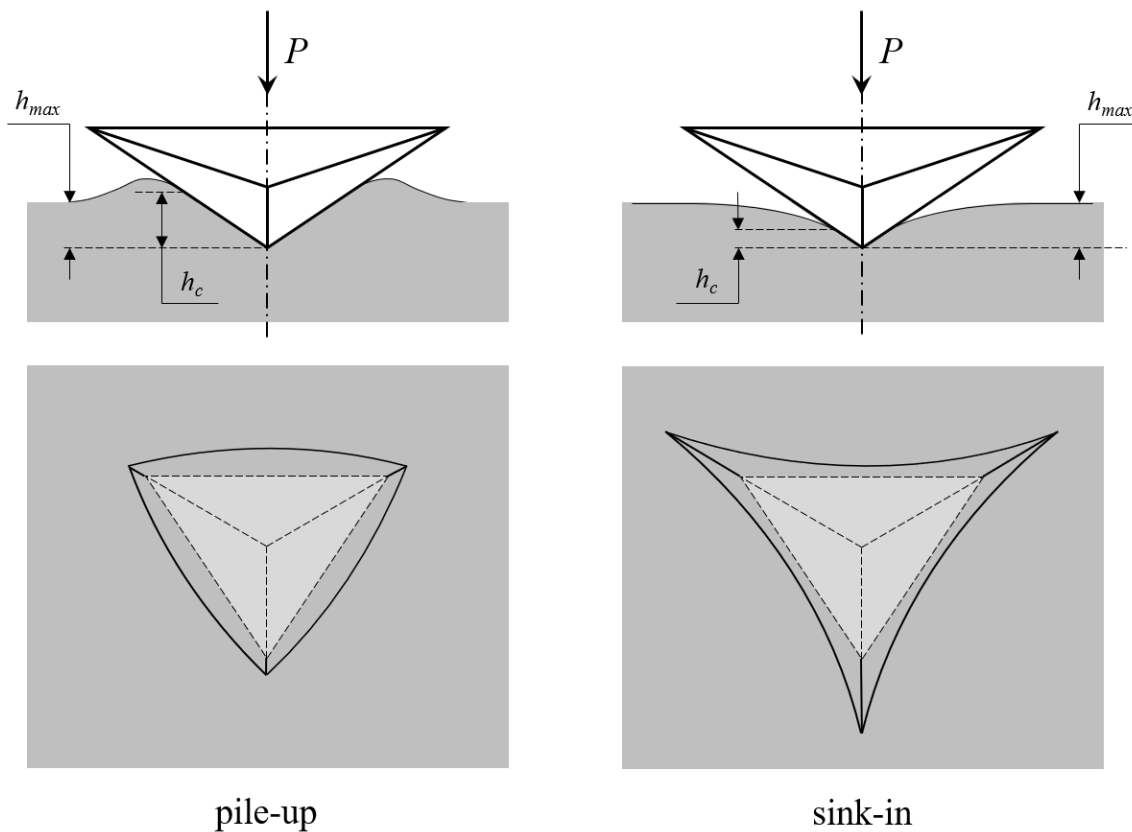


Fig. 1. Schematic illustration of indentation with pile-up and sink-in.

where $S = dP/dh$ is the contact stiffness and ε is a coefficient that depends on indenter geometry. For an ideal Berkovich indenter, the contact area is related to the contact depth by a quadratic relationship:

$$A_p = 24.56h_c^2. \quad (3)$$

In an actual experiment, the indenter tip has a finite radius and deviates from ideal geometry. An experimentally determined area function is therefore used.

The elastic modulus is determined through the reduced modulus:

$$1/E_r = (1-\nu_s^2)/E_{IT} + (1-\nu_i^2)/E_i, \quad (4)$$

$$E_r = \sqrt{\pi A_p} \times S/2, \quad (5)$$

where E_{IT} and ν_s are the elastic modulus and Poisson's ratio of the tested material, respectively, while E_i and ν_i are the corresponding properties of the indenter material.

Because a diamond indenter has a very high elastic modulus, the specimen provides the dominant contribution to the reduced modulus in the case of aluminum. When processing experimental data, it should be borne in mind that the ideal area relationship is an approximation. Oliver and Pharr discussed in detail the influence of actual indenter geometry and the refinements required for correct property determination [22].

2.2. Pile-up and sink-in at the contact

One of the most important features of indentation of metals is the discrepancy between the geometrically calculated contact area and the actual contact area. In plastically deformable materials, material may pile up around the indentation, as shown in Fig. 1. In this case, the surface rises around the indenter faces, causing the actual contact area to exceed the area calculated from contact depth alone. Using finite-element modeling, Bolshakov, Oliver, and Pharr showed that pronounced pile-up can lead to a substantial error in hardness determination if the actual contact area is not considered [23].

The opposite phenomenon is surface sink-in. In this case, the material surface around the indentation is displaced downward relative to the original surface. The actual contact area is smaller than in the case of pronounced pile-up. For soft materials capable of substantial plastic deformation, accurate determination of the actual contact area is particularly important. The authors of Ref. [24] showed, using soft films on hard substrates as an example, that pile-up can substantially affect the interpretation of nanoindentation results. This issue is especially important for aluminum and aluminum coatings because aluminum can undergo considerable plastic deformation even at relatively shallow indentation depths. The effects of creep

and pile-up on nanoindentation results for aluminum metallization have already been investigated [25]. Therefore, for aluminum, the measured depth cannot be regarded as a direct equivalent of the contact depth; the relationship between the two depends on the deformation behavior of the material.

2.3. Strain hardening of aluminum

Aluminum alloys exhibit different capacities for strain hardening. During indentation, plastic deformation is localized within a relatively small material volume. Consequently, the resulting load–displacement curve reflects not only the initial hardness but also the ability of the material to increase its resistance to deformation as plastic strain develops. Study [26] demonstrated the applicability of a work-of-indentation approach to the characterization of 2024-T351 aluminum and aluminum cladding. This is particularly important for materials in which hardness and resistance to plastic deformation are associated with strain hardening. To assess the contribution of plastic deformation, the dependence of flow stress on plastic strain must be considered. In the simplest approximation, this dependence may be described by the Hollomon power law, $\sigma = K\epsilon^n$, where K is the strength coefficient and n is the strain-hardening exponent [27]. During indentation, a high strain-hardening exponent can change the shape of the plastic zone and the balance between pile-up and sink-in. Therefore, identical hardness values do not necessarily imply identical indentation behavior.

3. SPECIFIC FEATURES OF INSTRUMENTED INDENTATION OF ALUMINUM

3.1. Effects of pile-up, sink-in, and strain hardening

For aluminum, pile-up and sink-in must be considered together with strain hardening [28]. At a low degree of strain hardening, the plastic zone can spread relatively easily and material is displaced toward the perimeter of the impression, which may promote pile-up. With more pronounced strain hardening, resistance to further plastic flow increases, changing the geometry of the plastic zone and potentially promoting more pronounced sink-in. In Ref. [29], the mechanical properties of AA2024 aluminum alloy were determined using instrumented nanoindentation combined with deep-learning methods. The study demonstrates that the full shape of the indentation curve contains information not only on hardness but also on elastic–plastic material parameters. Pile-up in a TiAl alloy was investigated using a combination of nanoindentation and crystal-plasticity modeling [30]. Although this material differs from aluminum alloys, the methodological conclusion is relevant: indentation geometry should be

regarded as the result of the interaction among indenter geometry, plastic flow, and strain hardening.

The effect of microstructure on the properties of aluminum alloys is especially pronounced [31]. In Ref. [32], wrought aluminum alloys after high-speed milling were investigated, and the authors showed that technological processing changes the condition of the surface layer. The same group subsequently demonstrated a relationship between high-speed machining of aluminum alloys and material-deformation processes [33]. Consequently, when comparing indentation results obtained from surfaces subjected to different technological operations, not only roughness differences but also possible changes in the material state within the surface layer must be considered. This is particularly important for machined aluminum parts. If a surface simultaneously exhibits increased roughness and a work-hardened surface layer, changes in indentation results cannot be attributed solely to roughness.

3.2. Effect of surface roughness

Surface roughness creates an initial contact geometry that differs fundamentally from that of an ideal flat surface. At the same nominal load, the indenter may contact the crest of an asperity, a valley, or a region close to the mean surface level. The effect of surface roughness on mechanical properties determined by nanoindentation was specifically investigated in Ref. [34]. The authors showed that roughness can alter the determined properties and increase measurement uncertainty. The problem becomes even more pronounced for thin metallic films [35], where surface roughness affects indentation results because the roughness scale can become comparable with the indentation depth.

During indentation of a rough surface, the measured depth h is effectively referenced to the local surface position, whereas the data-processing algorithm interprets it according to the adopted contact-geometry model. As a first approximation, the measured depth relative to the mean surface level may be represented qualitatively as $h = h_{real} \pm h_{roughness}$, where the roughness-related term reflects local deviation from the mean level h_{real} . This expression should be regarded only as a qualitative model. The actual error depends not only on asperity height but also on its spatial scale, shape, the position of the indent, and the nature of plastic deformation. If indentation occurs on an asperity crest, local contact $h(0)$ is established earlier relative to the mean surface level (Fig. 2). The local surface geometry may also promote additional material pile-up. As a result, the calculated depth and contact area differ from those for an ideal surface. When the indenter falls into a valley, the initial surface lies below the mean level; the depth referenced to the mean level may therefore be larger even though the local depth relative to the

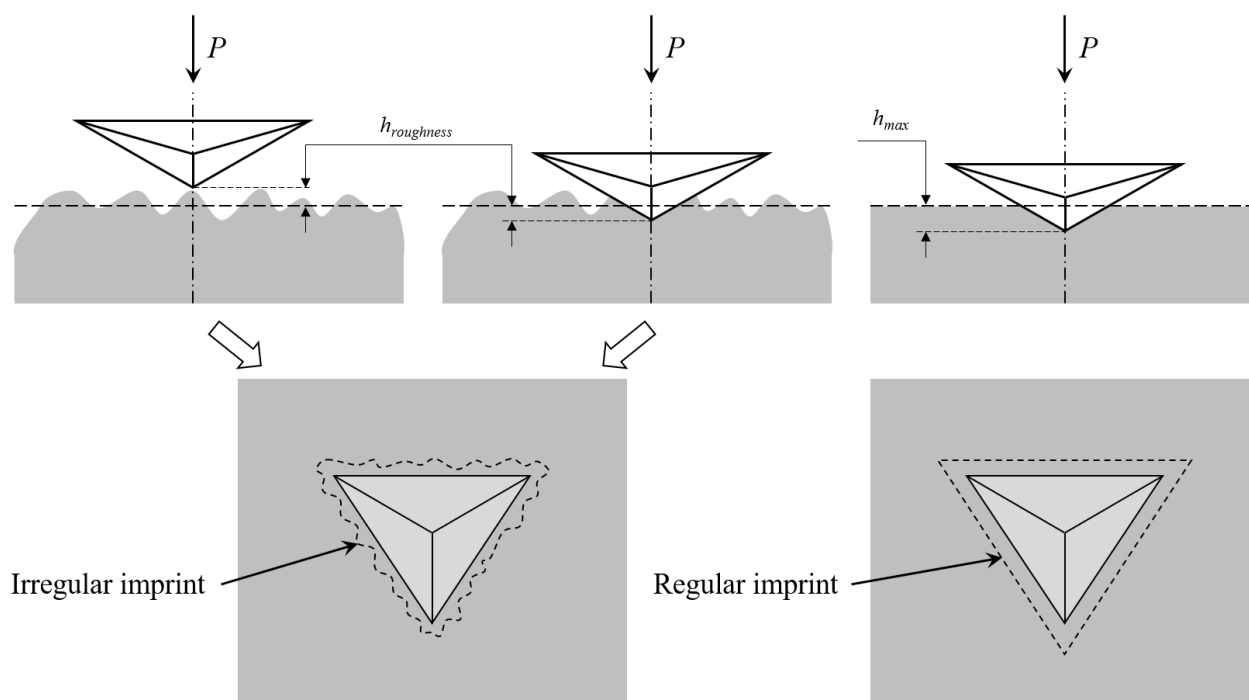


Fig. 2. Schematic illustration of indentation on a rough surface.

actual contacting surface is different. Thus, the same maximum load does not guarantee the same contact geometry.

This effect becomes particularly noticeable at shallow depths. If the indentation depth substantially exceeds the characteristic roughness height, the relative contribution of the surface microrelief to the error decreases. This is why increasing the load can sometimes reduce the relative effect of roughness. However, increasing the load is not a universal solution. Greater penetration increases the size of the plastic zone and, for thin coatings, may introduce a substrate effect.

3.3. Effect of specimen and coating thickness

For a bulk aluminum specimen, mechanical deformation in the indentation zone may be treated as occurring in a semi-infinite body. This assumption is no longer valid for a thin coating. When the indentation depth becomes comparable with the coating thickness, the substrate begins to contribute to deformation. As a result, the measured hardness and elastic modulus become effective properties of the coating–substrate system. This problem is particularly relevant for aluminum coatings on rigid substrates. For example, Ref. [36] examined errors in the determination of the properties of thin metallic films on hard substrates, including errors associated with pile-up. The aforementioned study [26] on AA2024-T351 also demonstrates the practical importance of local characterization of aluminum cladding. Therefore, for thin coatings, the ratio of indentation depth to coating thickness must be controlled.

At excessive penetration depths, the measured value cannot be interpreted as an intrinsic property of the aluminum layer.

3.4. Contact-area determination

One of the central issues is accurate determination of the contact area (or A_p). For an ideal geometric indenter, the area is uniquely determined by the contact depth h_c . In the presence of pile-up, however, the actual area is larger than the calculated value, whereas pronounced sink-in leads to a smaller area [37]. Using titanium as an example, the authors of Ref. [38] showed that simultaneous consideration of pile-up and sink-in is essential for determining the contact area. The same methodological conclusion applies to aluminum: calculation of hardness using an idealized area function may be insufficient when plastic deformation is pronounced.

Local loading of aluminum materials can generate regions of intense plastic deformation. Diffusion-bonded joints between aluminum foam and a magnesium alloy were investigated in Ref. [39], demonstrating the complex mechanical behavior of porous and bimetallic systems. Local properties of AA6061 after friction-stir welding and subsequent aging were investigated in Ref. [40], confirming the high sensitivity of indentation to local structural changes. A previously mentioned study examined an aluminum film in a Ni/Al surface system after ion-beam mixing [41], where the depth dependence of properties becomes particularly important. Deformation-band for-

mation during impact indentation of Al–6Mg was investigated in Ref. [42], showing that local indenter loading can generate pronounced spatial heterogeneity of plastic deformation. Size effects in continuous nanoindentation have also been investigated [43], demonstrating the importance of scale when aluminum materials are studied from the microindentation to the nanoindentation range. The use of indentation instruments for studying alloy surface properties was considered in Ref. [44], emphasizing the need to account for the actual condition of the tested surface during local characterization. The authors of Ref. [45] investigated the relationship between ultimate tensile strength and Brinell hardness for structural materials. Despite the difference in test methods and scales, this study is relevant to the broader problem of correlating local and bulk mechanical characteristics.

3.5. Other influencing factors

3.5.1. Technological surface condition

The surface condition of an aluminum component may change as a result of machining, polishing, finishing, and other technological operations. The authors of Ref. [46] demonstrated the importance of metallic-surface preparation for the service performance of components. Although that study focused on corrosion resistance, its methodological implication is relevant here: the surface is an independent characteristic of the object and should not be treated merely as a geometric boundary. The quality of complex-profile aluminum surfaces after magnetic-abrasive finishing has also been investigated [47,48]. These studies are relevant to indentation because changes in roughness caused by processing may be accompanied by changes in the structure and condition of the surface layer, including the surface oxide film [49]. Therefore, when polished and unpolished surfaces are compared, it is desirable to ensure an equivalent material condition within the indentation-affected depth.

3.5.2. Structural condition of aluminum

Study [50] examined microstructural features of Al–Yb master alloys produced by aluminothermic reduction. Such studies demonstrate the relationship between composition and structure of aluminum materials and their resulting mechanical properties. Indentation modeling was used to investigate the response of aluminum cladding panels to hailstone impact [51], extending indentation concepts from static property determination to modeling local deformation resistance. Study [52] investigated the effect of aluminum deformation conditions on microhardness and the indentation size effect (ISE), directly confirming the need to account for indentation scale when comparing

test results. The authors of Ref. [53] proposed estimating the mechanical properties of aluminum alloys using the indentation curve together with the projected area of the contact zone. This approach is of interest for increasing the information obtained from indentation without resorting to conventional tensile testing. The indentation size effect in aluminum and titanium alloys was investigated in Ref. [54]. For aluminum, this is especially important because changing the indentation size may change the measured hardness.

3.5.3. Mechanical properties

The authors of Ref. [55] investigated deformation-induced structuring of aluminum films during microindentation, confirming that indentation itself can modify the structure of the tested surface layer. In Ref. [56], the specific deformation behavior of an AlN–D16T topocomposite during instrumented indentation was investigated. The study shows that, for complex multiphase systems, standard interpretation of the load–displacement curve may be insufficient. The effect of aluminum-powder particle size on the structural-phase composition and properties of Al_2O_3 –AlN coatings was investigated in Ref. [57]. Local indentation is particularly informative for such materials because of their heterogeneous structure. The structure and physical-mechanical properties of ceramics based on aluminum and zirconium oxynitrides were investigated in Ref. [58]. This work is relevant to aluminum-containing materials but also emphasizes the need to distinguish clearly between the properties of metallic aluminum and aluminum-containing ceramics. The authors of Ref. [59] examined the effect of hydrogenation on the hardness of metallic materials, confirming the general sensitivity of local hardness to prior physicochemical exposure. The authors of Ref. [60] considered the determination of material characteristics from software-assisted instrumented-indentation results, an approach that is particularly promising for automated inspection. The influence of scale factor on the modulus of elasticity determined by instrumented indentation was demonstrated in Ref. [61]. This result is relevant to aluminum because methodological scale effects must be separated from true material differences when measurements obtained at different scales are compared.

3.5.4. Dynamic indentation and strain rate

Dynamic indentation extends the capabilities of the method by characterizing material behavior at increased loading rates. Dynamic hardness of structural metallic materials has been investigated in Ref. [62]. Although this approach differs from conventional quasi-static instrumented indentation, it demonstrates the influence of load-

ing regime on the measured characteristic. In Ref. [63], a nondestructive method for evaluating metal plasticity using a single impact microindentation was proposed. This approach is of particular interest for industrial inspection when rapid in-situ assessment of a component is required. Impact indentation was applied to the evaluation of cast-iron mechanical properties in Ref. [64], while Ref. [65] investigated the possibility of monitoring regions of local plastic deformation during fatigue wear of metallic structures using dynamic instrumented indentation. Although these studies are not limited to aluminum, they demonstrate the broader potential of instrumented indentation as a local diagnostic method. Studies directly addressing the effect of indentation strain rate on the properties of aluminum and related systems are also available [66–68].

Thus, the main factors and mechanisms by which surface roughness affects indentation results can be identified from this part of the review. These mechanisms are summarized in Table 1.

4. MECHANICAL PROPERTIES OF ALUMINUM DETERMINED BY INDENTATION

4.1. General characteristics

Aluminum alloys exhibit a wide range of mechanical properties; therefore, the use of a single reference hardness value for all aluminum grades is inappropriate. In Ref. [69], the authors demonstrated the use of instrumented indentation as a rapid method for evaluating the mechanical properties of aluminum alloys. This work is particularly important from the standpoint of industrial application because it demonstrates local characterization without the need to prepare standard mechanical-test specimens. For diffusion-bonded joints between aluminum and austenitic

stainless steel, nanomechanical properties and indentation creep were used to analyze the joint region in Ref. [70]. This confirms the applicability of the method not only to homogeneous alloys but also to locally heterogeneous objects. In Ref. [71], a cylindrical-indentation method was proposed for predicting the tensile properties of additively manufactured aluminum alloys. Such approaches extend local indentation from hardness determination toward estimation of tensile-related mechanical characteristics.

The local mechanical properties of a 6061-T6 aluminum weld obtained by instrumented indentation and microtensile testing were compared in Ref. [72]. Such comparisons are fundamentally important because they allow the information content of local methods to be assessed relative to conventional mechanical testing. Study [73] investigated the mechanical properties of aluminum alloys by instrumented indentation using Almigo Hard as a case study, demonstrating the ability of the method to obtain local mechanical parameters of structural aluminum alloys. As noted above, instrumented nanoindentation combined with deep learning was used to determine elastic–plastic parameters of AA2024 [29]. This approach shows that the information content of the experiment is defined not only by a single hardness value but also by the full shape of the indentation curve.

4.2. Local and basic properties of aluminum alloys

An important feature of aluminum alloys is structural heterogeneity. Depending on alloy grade and condition, the material contains grains, grain boundaries, intermetallic particles, and other microstructural constituents. In a study of high-purity aluminum [74], nanoindentation was used to analyze the effect of alloying elements on local mechanical properties. The results demonstrate the sen-

Table 1. Surface roughness related mechanisms affecting indentation results.

Mechanism	Physical quantity / process / deformation feature	Effect on results
Shift of the first-contact point	The indenter contacts a local asperity or valley rather than the mean surface level	Shift in the depth reference $h(0)$
Change in local contact geometry	The real surface differs from an ideal plane	Change in projected contact area A_p
Local redistribution of plastic deformation	Surface relief changes the direction and distribution of material deformation	Changes in pile-up and sink-in
Change in indentation depth h	Part of the measured displacement is associated with the surface microrelief	Changes in h_{max} and h_c
Increase in measurement scatter	Random position of the indent relative to the surface microrelief	Reduced repeatability
Indentation onto an asperity	Contact begins earlier; material can be displaced outward more readily	Potentially more pronounced pile-up and a larger actual contact area
Indentation onto a valley	The indenter first penetrates a local depression; surrounding asperities restrict material flow	Potentially more pronounced sink-in and a change in contact depth h_c

sitivity of local hardness to chemical composition. Cyclic dynamic response and creep of pure aluminum were investigated by instrumented indentation in Ref. [75], showing that indentation can be used not only to determine static hardness but also to assess the dependence of material behavior on loading regime. In Ref. [76], scanning electron microscopy and in-situ nanoindentation were applied to anodic films on A356. This is particularly important for analysis of aluminum surface layers, whose properties can differ substantially from those of the substrate.

For spatially integrated characterization, high-resolution nanoindentation mapping was used in Ref. [77] to investigate heterogeneity in the mechanical response of an additively manufactured aluminum alloy. This approach shows that a mean hardness value can conceal substantial local variation. In Ref. [78], nanoindentation was used to study AA6061 reinforced with carbon nanotubes and graphene, demonstrating the applicability of the method to assessing the effect of dispersed reinforcing constituents. The authors of Ref. [79] used nanoindentation to investigate intermetallic particles in AA2024. This is important for interpretation because, at small indentation sizes, a measurement may characterize an individual microstructural constituent rather than the bulk alloy.

4.3. Coatings

Aluminum coatings constitute a separate class of test objects [25,80]. As noted above, these studies show that creep and pile-up must be considered simultaneously when aluminum metallization is investigated. At shallow indentation depths, these effects can substantially alter the calculated contact area. For thin films, the substrate effect is further compounded by surface roughness [35]. Therefore, when developing an indentation procedure for an aluminum coating, both a minimum depth sufficient to reduce the relative influence of roughness and a maximum depth below which the substrate effect remains insignificant must be specified [81]. A work-of-indentation approach can also be applied to aluminum cladding [26].

A separate class of objects comprises surface layers produced by ion-beam treatment [41]. In that study, the mechanical properties of nickel surface layers with a sputtered aluminum film after ion-beam mixing were investigated, showing that the measured response in such systems is directly related to the through-thickness structural gradient.

4.4. Aluminum as a reference material for calibration

Accurate determination of the actual indenter area function is required to obtain reliable hardness and elastic-modulus values. Aluminum is of interest as a model metallic material because its elastic and plastic properties

are relatively well characterized. However, the use of aluminum as a calibration material should not automatically be considered universally preferable: its own deformation behavior, pile-up/sink-in response, and surface roughness must also be controlled. Studies involving direct measurement of indenter geometry [6,7] show that calibration can be performed from the topography of the working surface. An alternative is experimental calibration of the area function using materials with known properties.

The authors of Ref. [8] demonstrated the possibility of reconstructing indenter-tip characteristics using machine-learning methods. This approach is particularly promising for monitoring changes in indenter geometry during long test series. Study [4] showed that even Berkovich indenters with nominally identical geometry can produce different hardness values. Consequently, comparison of results from different studies requires consideration not only of the indenter type but also of the specific indenter, its calibration, and the condition of its tip. For aluminum, this issue is further complicated by the fact that typical indentation depths may be on the order of hundreds of nanometers, i.e., in a range where the tip radius may already be comparable with the characteristic contact size.

4.5. Summary of published property data

A number of studies provide sufficiently detailed hardness and elastic-modulus values for aluminum alloys tested by indentation. Table 2 summarizes selected numerical values for different aluminum grades. It should be noted that some elastic-modulus values in the table were not obtained by indentation, while some hardness values were derived from microhardness measurements; therefore, the generic symbols H and E are used for values taken from those studies. These values are indicated in the table with the asterisk (*). For example, in Ref. [53], elastic modulus (66.84*) was determined from tensile testing for AA5052, AA6061, and AA7075 because the principal aim of that work was to determine yield strength. From Ref. [18] table presents the properties only for material that was aged at a temperature of 180 °C for 8 hours.. For commercially pure Al in Ref. [15], $H = 0.60 \pm 0.12$ GPa and $E = 71.09 \pm 4.63$ GPa were reported. These values are close in initial hardness, prior to surface modification, to the result $H = 0.508 \pm 0.019$ GPa obtained in Ref. [14]. In Ref. [14], which investigated AA1100 and an aluminum coating, the initial AA1100 had $E = 68.48$ GPa, while after coating formation, it became $E = 69.99$ GPa. The authors specifically noted that a substantial increase in hardness after cold deformation ($H = 2.03 \pm 0.66$ GPa) was accompanied by almost no change in elastic modulus. Since the reported coating thickness was below 1 μm , an additional influence of the coating-substrate system on the nanoindentation response cannot be entirely excluded. In the

Table 2. Hardness and elastic modulus values for aluminum and its alloys

Material	H , GPa	E , GPa	P_{max} , mN	h_{max} , nm	No. of measurements / statistics	Ref.
A356	0.6 ± 0.1	–	500	–	–	[76]
AA1100 (as-recieved)	0.508 ± 0.019	–	1	≈ 200	mean	[14]
AA1100 (friction-surface coating)	2.03 ± 0.66	–	1	≈ 200	mean	[14]
AA2024	–	64.3	300	< 3000	29	[29]
AA2024-T3	2.00 ± 0.12	76.06 ± 2.91	1000	–	mean	[15]
AA5052	–	66.84*	< 110	2000	–	[53]
AA5083-H116	1.31 ± 0.04	75.34 ± 1.36	1000	–	mean	[15]
AA5083-H116	1.98	–	1	≈ 130	69	[17]
AA5083-H116	1.41	–	40	–	12	[17]
AA5083-H116 (deformed)	≈ 2.2	–	40	–	mean	[17]
AA6060	0.73 ± 0.02	70	800	< 9000	10	[18]
AA6061	–	67.66*	< 160	2000	–	[53]
AA6061	≈ 1.1	–	8	508	–	[10]
AA7075	–	68.44*	< 180	2000	–	[53]
AA7075-T651	2.23 ± 0.13	72.28 ± 1.98	1000	–	mean	[15]
Al-10V	6.24 ± 0.23	108.53 ± 3.62	1000	–	mean	[15]
Al-2V	2.43 ± 0.11	88.14 ± 2.79	1000	–	mean	[15]
Al-5V	5.21 ± 0.36	96.21 ± 2.47	1000	–	mean	[15]
Pure Al	0.60 ± 0.12	71.09 ± 4.63	1000	–	mean	[15]
Pure Al	0.21 ± 0.02	–	< 10	< 900	64	[82]
Pure Al	0.25 ± 0.05	70 ± 5	250	< 3000	–	[75]

local AA1100 measurements reported in Ref. [13], elastic modulus of $E = 70.65 \pm 1.93$ GPa was obtained in the region with lower local mechanical heterogeneity, whereas regions near particles yielded $E = 74.60 \pm 1.76$ GPa. A reference value of approximately $E = 68.9$ GPa for AA1100 was also reported.

The situation is more complicated for hardness. In Ref. [82], $H = 0.21 \pm 0.02$ GPa is compared with a microhardness value of $HV = 0.198 \pm 0.002$ GPa. Thus, considering the pile-up effect, the hardness reported for commercially pure aluminum can be regarded as substantially underestimated. In a more recent study [11] on surface-modified AA1100, the initial aluminum exhibited $H = 0.12 \pm 0.02$ GPa, whereas local hardness near grains in AA1100 could reach $H = 0.98 \pm 0.08$ GPa.

Published data show that the mechanical properties of aluminum and its alloys cannot be reduced to a single universal value. For high-purity aluminum, elastic-modulus values are expected to be close to the reference level of approximately $E = 70$ GPa, although local measurements may deviate because of structural state, the indentation-curve processing method, and uncertainty in contact-area determination. For aluminum alloys, the elastic modulus is also generally close to this range, whereas hardness may vary much more substantially. This difference arises because elastic modulus is governed primarily

by interatomic bonding, while hardness is much more sensitive to alloying, precipitation strengthening, grain size, heat treatment, and strain hardening. Therefore, when interpreting an experimentally measured hardness value for an aluminum alloy, not only the alloy grade but also its condition, processing history, and indentation conditions should be specified. As a practical reference, measured values for pure aluminum may be taken as $H_T = 0.1 \dots 1.2$ GPa and $E_T = 68 \dots 76$ GPa. For alloys containing strengthening additions, substantially higher hardness values may be obtained.

5. EXPERIMENTAL EVALUATION OF HARDNESS AND MODULUS OF AN ALUMINUM ALLOY AT DIFFERENT SURFACE ROUGHNESS LEVELS

5.1. Experimental procedure

To assess the applicability of instrumented indentation, the effect of surface roughness on indentation results was investigated. Four specimens of commercially produced AMg2 aluminum alloy (broadly comparable in composition to AA5052) were used. Each specimen had in-plane dimensions of 2×20 mm and 5 mm thick. The test surface of each specimen was evenly divided into 2×10 mm re-

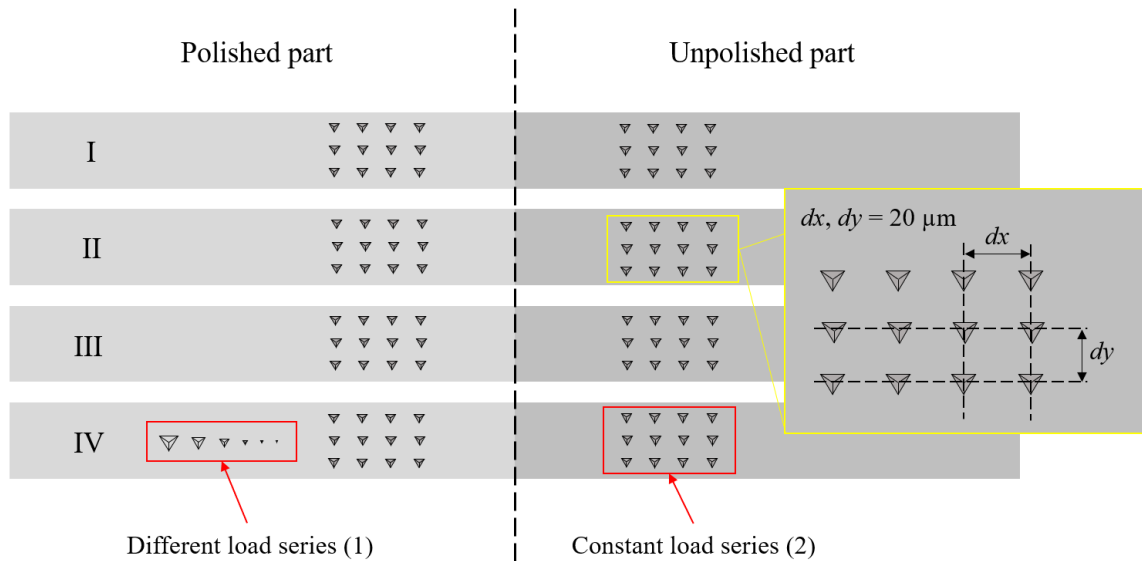


Fig. 3. Layout of indentation locations on the specimens.

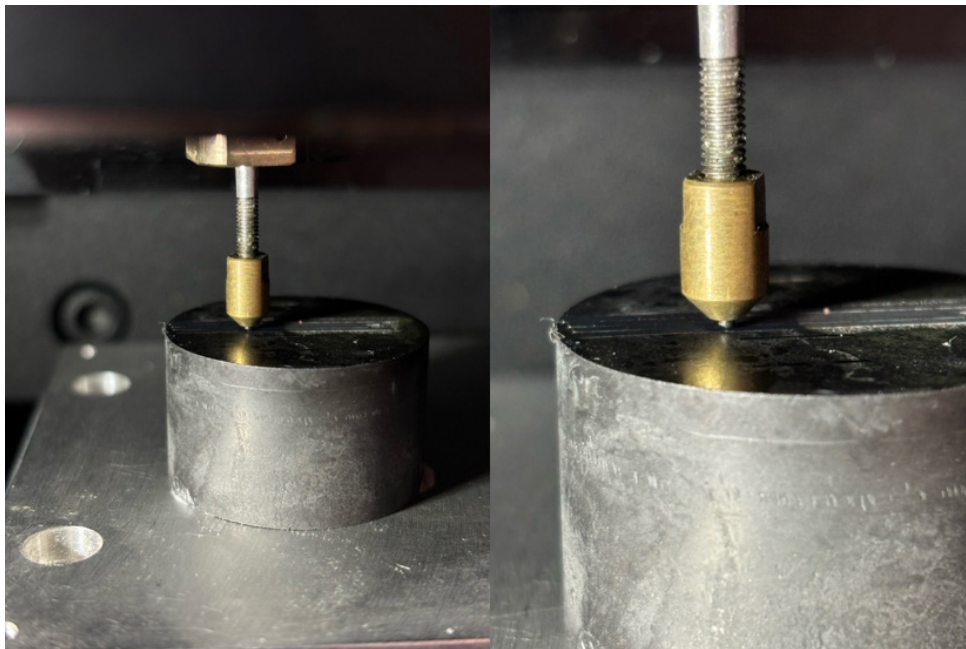


Fig. 4. Images of the specimen under the indenter during testing.

gions with different roughness. This alloy is one of the widely used aluminum alloys and has also been extensively characterized in the literature [83,84]. Each specimen contained two surface regions: polished and unpolished. The mean roughness was $Ra = 0.041 \mu\text{m}$ for the unpolished region and $Ra = 0.029 \mu\text{m}$ for the polished region. Surface roughness was measured using a surface roughness tester Hommel Tester T1000 and for each surface condition, Ra was determined from four independent profiles/areas. The resulting values for both polished and unpolished had a coefficient of variation (CV) less than 7%. The properties measurement layout is shown in Fig. 3. Preliminary tests with different loads were carried out on the polished region of one specimen to select

the load for the main test series. During each preliminary test, the load on the indenter increased linearly. Each main test series then comprised 12 measurements at the same load, arranged according to the specified indentation matrix with $dx = dy$. The test conditions were as follows: $P_{max} = 1 \dots 200 \text{ mN}$ in the preliminary series, loading time $t_l = 5 \text{ s}$, and hold time $t_h = 1 \text{ s}$. The load for the main series was selected on the basis of the preliminary variable-load tests. Measurements were performed using a NanoScan 4D+ nano-hardness tester equipped with a diamond Berkovich indenter with Poisson's ratio $\nu = 0.17$ and elastic modulus $E = 1140 \text{ GPa}$ [85]. Calibration was performed using polycarbonate with known mechanical properties: $H_{IT} = 0.21 \pm 0.02 \text{ GPa}$ and $E_{IT} = 3.0 \pm 0.3 \text{ GPa}$.

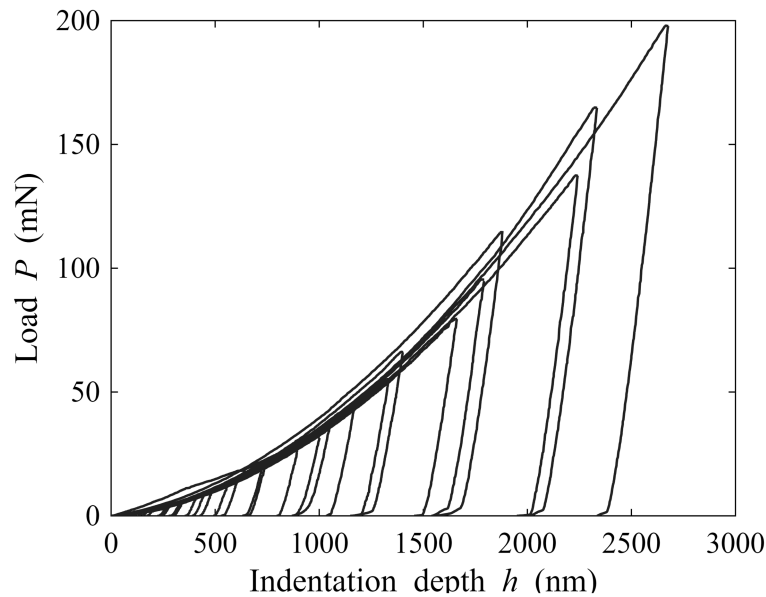


Fig. 5. Indentation curves $P(h)$ obtained at different loads.

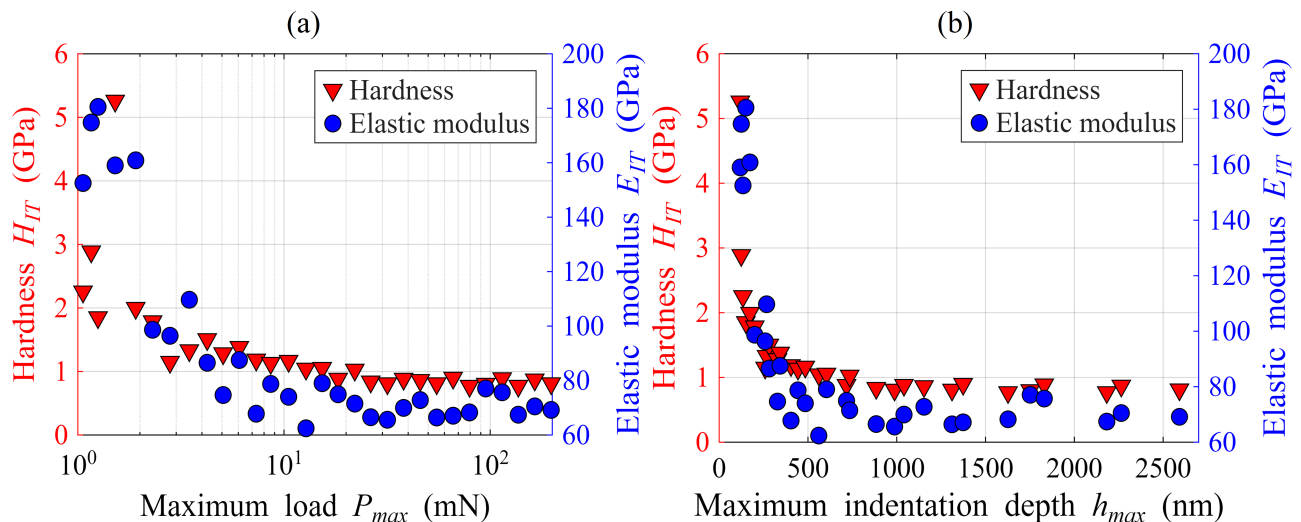


Fig. 6. Indentation results for tests at different loads: hardness and elastic modulus as functions of (a) test load P_{max} and (b) indentation depth h_{max} .

The appearance of the specimen during testing is shown in Fig. 4.

5.2. Results

The indentation curves $P(h)$ obtained during the preliminary tests with increasing load are shown in Fig. 5.

The hardness and elastic-modulus values calculated from the test data using the Oliver–Pharr methodology [21,22] are presented in Figs. 6a,b.

The preliminary tests established $P_{max} = 20$ mN as the load for the main test series because the mechanical-property values became stable above this level. It was also noted that, for a surface roughness of $0.029 \mu\text{m}$, Standard [86] recommends an indentation depth of at least

580 nm. In accordance with Section 5.1, 12 measurements were performed in each main series. Examples of indentation curves for these tests are shown in Fig. 7. A slight reduction in indentation depth was observed for the polished specimens, which may increase the calculated hardness.

After testing, the indentations were visually inspected to reduce the influence of obvious surface defects. An example image of the surface is shown in Fig. 8. The results of the main test series are presented in Tables 3 and 4.

The hardness of the polished specimens was found to be slightly higher: the mean value for all polished specimens was $H_{IT} = 0.959 \pm 0.016$ GPa (CV = 1.6%), while for the unpolished specimens $H_{IT} = 0.865 \pm 0.039$ GPa (CV = 4.5%). The coefficient of variation of hardness was therefore higher for the unpolished specimens. Specimen 4 is the most il-

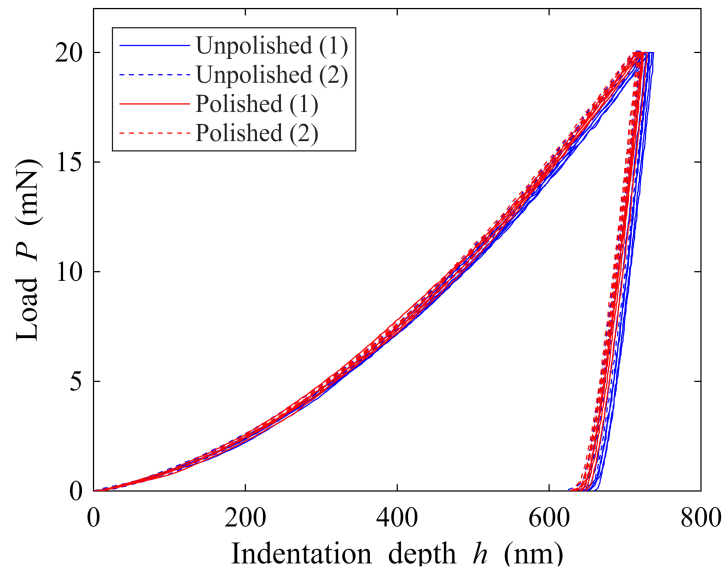


Fig. 7. Indentation curves $P(h)$ obtained at a constant load $P_{max} = 20$ mN.

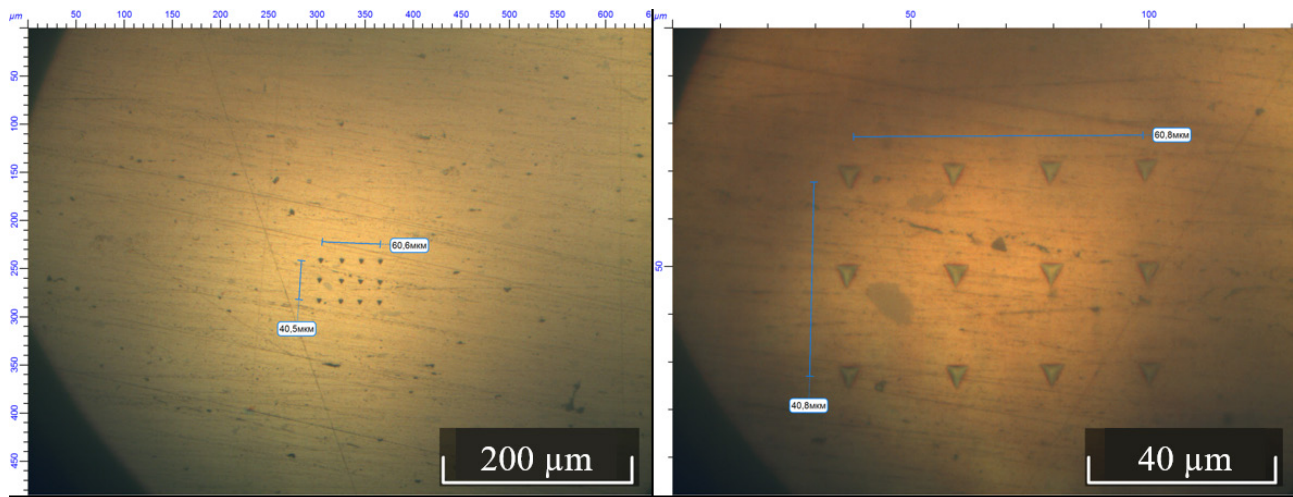


Fig. 8. Appearance of indentations on the test surface (polished surface).

Table 3. Results obtained at the same load (indentation hardness H_{IT}).

Specimen	Indentation hardness H_{IT} , GPa	
	Polished surface $Ra = 0.029 \mu\text{m}$	Unpolished surface $Ra = 0.041 \mu\text{m}$
1	0.957 ± 0.054	0.890 ± 0.062
2	0.954 ± 0.067	0.824 ± 0.057
3	0.981 ± 0.059	0.899 ± 0.061
4	0.943 ± 0.035	0.839 ± 0.146

Table 4. Results obtained at the same load (indentation elastic modulus E_{IT}).

Specimen	Indentation elastic modulus E_{IT} , GPa	
	Polished surface $Ra = 0.029 \mu\text{m}$	Unpolished surface $Ra = 0.041 \mu\text{m}$
1	76.21 ± 3.64	73.87 ± 3.64
2	73.88 ± 3.44	70.84 ± 3.34
3	77.15 ± 2.48	72.75 ± 3.49
4	76.09 ± 2.61	72.64 ± 7.71

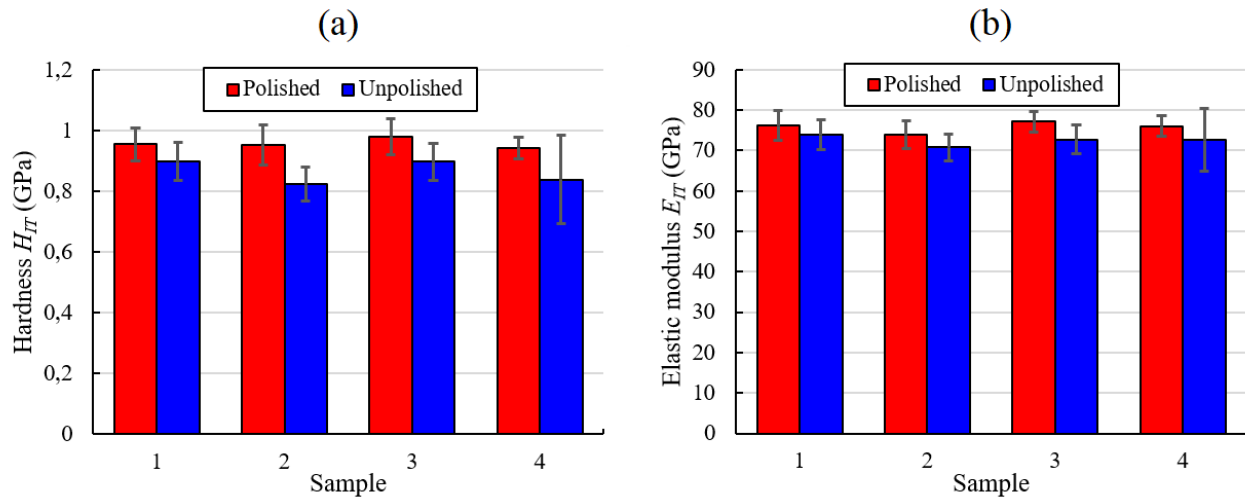


Fig. 9. Indentation results obtained at a constant load $P_{max} = 20$ mN: (a) hardness; (b) elastic modulus.

lustrative example: the scatter on the polished surface was 0.035 GPa and increased to 0.146 GPa on the unpolished surface. Thus, the absolute scatter increased by more than a factor of four, indicating that the principal manifestation of roughness in the present experiment is an increase in the uncertainty of an individual measurement. For the indentation modulus, the values averaged over all specimens were $E_{IT} = 75.83 \pm 1.38$ GPa (CV = 1.8%) and $E_{IT} = 72.52 \pm 1.25$ GPa (CV = 1.7%) for the polished and unpolished specimens, respectively. A graphical representation of the results with error bars as standard deviation is shown in Fig. 9. Thus, the indentation modulus changes primarily in its absolute level between the polished and unpolished surfaces, whereas the contribution of roughness within the investigated range is relatively small.

5.3. Analysis

5.3.1. Mechanical properties of aluminum.

The obtained hardness values are consistent with microhardness values of 80...97 HV reported in Ref. [87], 55.3...94.5 HV in Ref. [88], and 68...105 HV0.5 in Ref. [89]. They are also consistent with the range of 64...105 HV reported in Ref. [90]. Studies [83,84,91] report 59...101 HV, 60...116 HV, and 79...101 HV, respectively, which are also consistent with the present value $H_{IT} = 0.912 \pm 0.057$ GPa. The hardness of 0.96 GPa obtained for the polished surface falls within the range typical of relatively soft aluminum alloys. However, hardness alone is insufficient for unambiguous identification of an aluminum-alloy grade by indentation. This agrees with studies showing that aluminum-alloy hardness can vary substantially with structural condition. For example, pronounced local heterogeneity in AA2024 caused by in-

termetallic particles was demonstrated in Ref. [79]. Local properties of AA6061 likewise depend on structural condition and processing history [72,78].

The indentation elastic modulus determined in the present work $E_{IT} = 74.18 \pm 2.15$ GPa, lies within the ranges $E = 69.5...82.0$ GPa reported in Ref. [90] and $E = 45...83$ GPa reported in Ref. [92] for the comparable AA5052 aluminum alloy. Studies [16,74] on high-purity aluminum and AA6061 show that elastic-modulus values determined by instrumented indentation can be close to reference values, while a specific measured value depends on the method used to process the unloading portion of the $P(h)$ indentation curve. Studies [53,54] demonstrated the presence of the indentation size effect in different aluminum alloys and its influence on the measured properties. Therefore, comparison of the present results with literature data requires consideration not only of nominal load but also of actual indentation depth; for this reason, depth data are included in Table 2.

5.3.2. Effect of roughness.

Overall, the results allow the influence of roughness to be divided into two components: a change in the mean measured value and an increase in statistical scatter. For hardness, the mean value decreased by approximately 10% when the surface condition changed from polished to unpolished. However, this change must be considered together with within-series scatter; therefore, a conclusion regarding a stable systematic shift requires additional experiments with a larger number of measurements and quantitative characterization of surface roughness. The systematic change in elastic modulus is smaller, approximately 4%, and the measured values remain close to the expected range for aluminum. Thus, the present results are consistent with the view that roughness primarily re-

duces the repeatability of local contact conditions. This conclusion agrees with Ref. [34], which relates variations in nanoindentation results to surface roughness. The different sensitivities of hardness and elastic modulus are explained by their different dependence on contact area. Hardness is defined directly as the ratio of load to contact area according to Eq. (1); therefore, even a relatively small error in the projected contact area A_p directly affects the calculated hardness. Elastic modulus is determined from contact stiffness and has a more complex dependence on the unloading-curve parameters, so a local change in contact geometry caused by roughness does not necessarily produce a proportional change in E_{IT} . In the present experiment, preliminary tests showed stabilization of the measured properties from approximately 10 mN and an indentation depth of about 500 nm. If the characteristic asperity height remains constant, the ratio Ra/h decreases as indentation depth increases, reducing the relative contribution of roughness to the measured contact depth.

5.3.3. Possible effect of polishing on the near-surface layer:

Mechanical polishing can influence indentation results through two competing mechanisms. First, removal of asperities reduces geometrical uncertainty in the initial contact and improves the reliability of contact-area determination. At the same time, abrasive interaction during polishing can introduce local plastic strain into the near-surface layer of a ductile aluminum alloy, leading to an increase in dislocation density, residual stresses, and local strain hardening. Conversely, removal of a previously work-hardened surface layer may reduce the measured hardness, depending on the initial material condition and polishing procedure. Therefore, the approximately 9.8% higher mean indentation hardness measured on the polished regions in the present study cannot be attributed exclusively to the reduction in Ra . A contribution from polishing-induced modification of the near-surface microstructure is also possible. Since the subsurface dislocation structure and residual stresses were not independently characterized in the present work, these contributions cannot be quantitatively separated. Further investigation using EBSD, X-ray residual-stress measurements, or depth-resolved indentation would be useful for distinguishing the topographical and microstructural effects of polishing.

5.3.4. Effect of pile-up/sink-in.

Surface roughness is not itself the cause of pile-up or sink-in. However, it changes the initial contact geometry and can thereby affect development of the plastic zone. If the indenter contacts an asperity, contact begins at a locally elevated surface. During loading, material may be

displaced laterally and form additional pile-up. The actual contact area may then exceed the area calculated from ideal geometry. When the indenter is located in a valley, the situation differs: the initial contact geometry is shifted relative to the mean surface level and the local surface may exhibit pronounced sink-in. Therefore, the sequence “roughness $\rightarrow$ change in local geometry $\rightarrow$ change in depth $\rightarrow$ change in calculated area $\rightarrow$ change in hardness” provides a more appropriate physical model than the simple statement that roughness either increases or decreases hardness. Increasing the load also increases the plastic-zone size and may intensify pile-up. For thin coatings, it additionally increases the risk of substrate influence. It is therefore concluded that the optimum load should be selected not only to provide a stable hardness value but also by jointly considering surface roughness, specimen thickness, and plastic-deformation behavior.

On the basis of the reviewed studies and the present experimental results, the following evidence-based recommendations can be formulated:

- 1) A preliminary series at different loads should be performed before the main measurements in order to identify the range in which the measured properties become stable.
- 2) Both the maximum load and the actual indentation depth should be recorded. For aluminum and other soft alloys susceptible to pile-up, comparison of results obtained at different loads without accounting for depth may be incorrect.
- 3) When pronounced surface roughness is present, a larger number of indentations should be performed. In the present study, 12 measurements were used for each series, allowing within-series scatter to be evaluated.
- 4) Surface roughness and the condition of the surface layer should be monitored separately. If the surface has been mechanically processed, changes in the measured result cannot automatically be attributed to roughness alone.
- 5) For thin aluminum coatings, indentation depth should be limited relative to coating thickness.
- 6) When comparing data obtained with Berkovich indenters, the specific area function and the condition of the indenter tip should be considered.

6. CONCLUSIONS

The analysis performed in this work shows that instrumented indentation is a promising method for local determination of the mechanical properties of aluminum and its alloys. Its main advantages are the small test area, the ability to characterize heterogeneous materials and surface layers, and the possibility of obtaining both plastic and elastic characteristics.

For aluminum materials, indentation results are governed by a combination of factors. The most important

are actual indenter geometry, strain hardening and plastic-flow behavior, pile-up and sink-in, surface roughness, the indentation size effect, strain rate, and, for coatings, substrate influence.

Accurate determination of the actual contact area is particularly important. In the presence of pronounced pile-up, the ideal geometric area function may lead to systematic errors in hardness determination, resulting in either overestimation or underestimation. Therefore, when ductile aluminum materials are investigated, the morphology of the residual impression should be monitored or methods that directly assess contact-zone geometry should be applied.

The experimental study of the aluminum alloy showed that at a load of 20 mN the hardness values for the polished surface ranged from 0.943 to 0.981 GPa, while the elastic-modulus values ranged from 73.88 to 77.15 GPa. For the unpolished surface, the corresponding ranges were 0.824–0.899 GPa and 70.84...73.87 GPa. The mean hardness decreased by 9.8% and the mean elastic modulus by 4.4% when changing from the polished to the unpolished surface. The most characteristic effect was an increase in the scatter of individual measurements, particularly for hardness.

The results show that the influence of roughness cannot be reduced to a simple systematic overestimation or underestimation of mechanical properties. It is more appropriate to regard roughness as a factor that changes local contact geometry and increases the uncertainty in contact-area determination. For practical characterization of aluminum materials, a preliminary load-selection procedure should be used, indentation series should be processed statistically, and indentation depth should always be recorded.

The results of the present study are consistent with published data on the effects of surface roughness and the indentation size effect on instrumented indentation results. A promising direction for further work is the quantitative determination of relationships between roughness parameters R_a and R_z , the characteristic spacing of surface irregularities, and the relative error in hardness and elastic-modulus measurements for aluminum alloys.

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Анализ применения метода инструментального индентирования для алюминия и его сплавов

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Аннотация. В настоящей работе рассмотрены особенности применения метода инструментального индентирования к алюминию и алюминиевым сплавам. Основное внимание уделено влиянию эффектов навала и прогиба, деформационного упрочнения, шероховатости поверхности, толщины исследуемого слоя и геометрии индентора. Проанализированы опубликованные результаты исследований чистого алюминия, алюминиевых сплавов, алюминиевых покрытий и соединений, а также работы, посвященные совершенствованию процедур обработки диаграмм индентирования. Представлены результаты экспериментального исследования алюминиевого сплава марки АМг2 (AA5052). Показано, что при выбранной нагрузке 20 мН средние значения твердости и модуля упругости для полированной поверхности составили соответственно около 0.959 и 75.8 ГПа, тогда как для неполированной поверхности – 0.865 и 72.5 ГПа. При этом наиболее существенным проявлением шероховатости оказался не столько систематический сдвиг среднего значения, сколько увеличение разброса результатов, особенно для твердости. Полученные результаты сопоставлены с литературными данными и рассмотрены с точки зрения применимости инструментального индентирования для контроля алюминиевых материалов.

Ключевые слова: инструментальное индентирование; алюминиевые сплавы; твердость; модуль упругости