



Article The Relationship between Polishing Method and ISE Effect

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Abstract: The aim of the submitted work is to study the relationship between the method of polishing the metallurgical surface and the indentation size effect (ISE). The material of the sample was annealed 99.5% aluminum. The polishing time ranged between 300 and 3600 s. An aqueous emulsion of aluminum oxide (spineline) and diamond paste were used as the polishing agents. The surface quality of the samples was measured with roughness meters. Applied loads in the micro-hardness test were 0.0981, 0.2452, 0.4904, and 0.9807 N. The effect of polishing on micro-hardness, Meyer's index n, and ISE characteristics was evaluated using the PSR method and the Hays–Kendall approach. As the polishing time increases, the micro-hardness values decrease, and the value of Meyer's index n increases from "normal" to neutral, i.e., Kick's law applies. The finding was confirmed for both of the used polishing agents.

Keywords: aluminum; polishing; micro-hardness; ISE

1. Introduction

An indentation size effect (ISE), in general, is observed in shallow indentation tests of (micro-)hardness, which is manifested as an increase ("normal") or decrease (RISE) in hardness with penetration depth decreases.

Among other factors, the size and nature of the ISE are influenced by the sample preparation method (polishing time, polishing agent) to create the metallographic surface for micro-hardness measurement. The quality of the obtained surface is usually determined subjectively, so the goal is to achieve a mirror finish without visible scratches. The polishing process must be controlled so that the Beilby layer does not form. Although the quality of the metallographic surface did not change significantly during the polishing time in the observed range, this time had a statistically significant effect on the size, nature, and other parameters of the ISE. Thus, the polishing time, when inappropriately chosen, can distort the measurement results to a certain extent.

The measurement of micro-hardness makes it possible to determine the basic mechanical properties of a small volume of material using an almost non-destructive method. If, as in the case of (macro-)hardness, the Vickers method is used, the only difference is a lower load (lower than 1.691 N). Measurements of micro-hardness can be used for miniature components, thin surface layers, or a metallography. The shape of the indentation (pyramid) is geometrically similar for all test loads. It is therefore expected that the measured hardness will be over a broad load range if the tested sample is homogeneous.



Citation: Petrík, J.; Blaško, P.; Draganovská, D.; Kusmierczak, S.; Šolc, M.; Ťavodová, M.; Mihaliková, M. The Relationship between Polishing Method and ISE Effect. *Crystals* **2023**, *13*, 1633. https:// doi.org/10.3390/cryst13121633

Academic Editors: Umberto Prisco and Pavel Lukáč

Received: 30 September 2023 Revised: 6 November 2023 Accepted: 22 November 2023 Published: 25 November 2023



Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). Unfortunately, this statement only applies to the range of loads intended for measuring (macro-)hardness. If "a very low" test load is used, the measured value is influenced by other factors. However, the term "very low load" is not exactly defined. Standard ISO 6507-1:2018 lists loads (test forces) in the range between 0.009807 N (1 g) and 0.9807 N (100 g) [1]. However, according to standard ISO 14577-1:2015, the values of the loads for the micro-hardness tests are less than 2 N (~200 g), while the indentation depth h > 0.2 μ m [2]. As stated Voyiadjis and Peters, "a very low" load results in indentation with a depth less than 10 μ m (... but not less than 0.2 μ m in ISO 14577-1:2015) [2–4].

The ISE may be caused by the following:

- 1. The testing equipment—it includes the characteristics of devices used to measure the dimensions of indentations and loads [4–6].
- Intrinsic properties of the samples—work hardening during indentation, the load to initiate plastic deformation, the indentation elastic recovery and elastic resistance of the materials [5–7], and the influence of crystallographic orientation [8,9].
- 3. The method of preparing the samples—the cutting, the grinding, the polishing, and stresses in the samples resulting from their manufacture as well as many other factors such as the indenter/sample friction, the lubrication, the corrosion, and speed of the indenter's penetration [4,6,7,10,11].

In contrast to a "normal" ISE, a reverse (RISE) type of ISE, where the apparent microhardness increases with increasing test load, is also known. It mostly takes place in materials with predominant plastic deformation. As a rule, it is explained by the existence of a distorted zone near the crystal–medium interface, the effects of vibration and the bluntness of the indenter, the applied energy loss as a result of specimen chipping around the indentation, and the generation of the cracks [7].

As mentioned above, the quality and method of preparation of the metallographic surface, on which the measurement will be carried out, has an influence on the measured micro-hardness values and at the same time on the ISE parameters.

The mutual relationship between the quality of the measured surface, expressed by its roughness (for example, Ra), and the obtained values of micro-hardness (or nano-hardness) has already been addressed by several researchers in the past. As an example, ref. [12] studied the influence of the surface quality of nickel samples on hardness and, using the Nix–Gao model, derived the relationship between surface quality and critical contact depth. In [13], SCM21 steel samples were also analyzed in a similar way.

Xia et al. published the results of studying the impact of roughness on hardness for Ti alloy $AlTi_6V_4$, and Xia published the results of studying the impact of roughness on hardness for Ti alloy, without evaluating the impact on the ISE [14].

The relationship between the quality of the surface and the hardness of non-metallic materials (aluminum oxide and polystyrene) was studied [15]. The variation of Knoop micro-hardness (loads between 200 g/1.96 N and 1000 g/9.81 N) follows the reverse ISE trend, i.e., an increase in hardness on load in the low-load region beyond where it becomes relatively constant.

The influence of the surface roughness on the ISE in micro-indentation was examined using the proportional specimen resistance model [16]. Stainless steel, aluminum (6061-T6: 95.9–98.6% Al with 0.8–1.2 Mg and 0.4–0.8 Si), and copper surfaces were polished to different levels of roughness with spineline (alumina powder 5–0.05 μ m) and subjected to HV micro-indentation. The load ranged between 0.147 and 1.962 N. To evaluate the factors of material elasticity and friction effect that make up the elastic proportional resistance, coefficient a₁, related to the elastic properties (it characterizes the load dependence of micro-hardness and describes the ISE in the PSR model), was plotted against the sample surface roughness Ra. As the roughness increases, the value of this coefficient for all three tested materials increases. As mentioned below, a similar relationship between roughness and parameter a₁ was observed by the authors of the paper. To evaluate the roughness effect on the ISE, an equation to predict the ISE was proposed, corresponding to the surface roughness factor for micro-indentation.

Another important area in the relationship between roughness and micro-hardness is the resin composite used in dental medicine, polished by one-step polishing systems or by a conventional multi-step system, studied, for example, by Edemir et al. or Korkmaz et al. [17,18]. As in the case of work focused on metal materials, for these materials, the authors focused only on the relationship of roughness–(micro-)hardness, without further study of the ISE.

As a follow-up to the mentioned works, the authors of the present contribution tried to influence the method of polishing the metallurgical surface and its surface roughness. They evaluate the impact of these factors not only on micro-hardness but also on the size and nature of the indentation size effect (ISE).

2. Materials and Methods

Tempered (400 °C/1 h) 99.5% Al (EN AW 1350) in form of the wire (diameter 9 mm) with yield strength (YS) = 25 MPa, ultimate tensile strength (UTS) = 73 MPa, total elongation (TE) = 59.7%, and the reduction of the area (or contraction Z) = 90.7% was an experimental material [19].

The wire was cut using a cooled diamond saw perpendicular to the axis. The pieces were in random order embedded in the resin (dentacryl) and ground with silicon-carbide papers in the sequences 80, 220, 240, 280, 500, 800, 1000, and 3000 ANSI/CAMI. The metallographic surface was subsequently polished 300, 600, 1200, 1800, 2400, 3000, and 3600 s with polishing agents by the same operator:

- 1. Aqueous emulsion of Al_2O_3 (alumina powder or spineline, 400 mL H₂O, and 25 g Al_2O_3 with grain size 10–40 µm); felt was used as a textile for the polishing wheel.
- Diamond paste (product by Pramet/Urdiamant Sumperk, Czech Republic) in the 2–3 μm size ranges (corresponding with the D2 FEPA Fédération Européenne des Fabricants de Produits Abrasifs) moistened with kerosene; velvet was used as a textile for the polishing wheel.

In both cases (spineline and diamond), the circumferential speed of the polisher disc (ø 270 mm) was 11 revolutions per second.

When viewed with the naked eye for both agents used for polishing, after 300 s of polishing, the surface was mirror-like, without visible scratches or grooves. On the contrary, at a magnification of $200 \times$, the scratches were visible regardless of the polishing time. Even polishing for 3600 s did not completely remove them. On the contrary, even with longer polishing times, the formation of the Beilby layer (over-polishing) was not observed [20]. This was probably the result of thorough wetting of the polishing wheel textile, the rotational movement of the samples, and a reasonably chosen pressing force (polishing was carried out manually, with all samples by one operator).

To objectify the quality of the ground (3000 ANSI/CAMI grit) and polished surface, its roughness Ra (arithmetical mean height; arithmetical mean height indicates the average of the absolute value along the sampling length) was measured using standard ISO 4287:1997 [21].

In the first stage, the roughness was measured with a contact tester Surftest SJ301 (Mitutuyo). N = 5 (5 sections of 0.5 mm each), i.e., the measured length l = 1.25 mm, and the radius of the sensor was 0.4 μ m.

As found using microscopic examination of the surface of the samples after measuring the roughness, the arm of the tester device left traces on the surface of the sample. It means that due to the low hardness of the samples, the compressive force caused plastic deformation. Therefore, we can consider the measured values of Ra as indicative at best, even though the value of W (the smallest load that causes an indentation) is in the range of 0.0019 to 0.0384 N, and the pressing force of the tester (measuring force) is 0.00075 N [22].

The test material was too soft to use a contact roughness meter. Therefore, in the following, we will consider the roughness values measured in this way only as indicative.

The mentioned deficiency was eliminated by measuring roughness with a non-contact confocal laser scanning microscope Olympus LEXT 3100. The hardness values of the

ground sample (3000 ANSI/CAMI grit) and the polished samples were measured in two mutually perpendicular directions (x-y). From the measured roughness values (e.g., Rp, Rv), the value of Ra was used in the next arithmetical mean height (Ra), which indicates the average of the absolute value along the sampling length.

A Hanemann tester, type Mod D32 fitted to microscope Neophot-32Micro-hardness, measured the micro-hardness with a magnification $480 \times$.

The tester meets the requirements of the standard ISO 6507-2, 2005, due to its repeatability $r_{rel} = 4.22\%$, an error of tester $E_{rel} = -0.92\%$, and the relative expanded uncertainty of calibration $U_{rel} = 6.76\%$ [23].

The values of the U_{rel} uncertainty of the measured micro-hardness of the sample listed in Table 1 are overvalued, considering the relationship between u_{CRM} (4.0 HV0.05) and the mean micro-hardness of the samples (29.3 for spineline and 26.1 for diamond); therefore, it should only be taken as an informative value.

Table 1. The values of polishing time, "the path of the sample on the polishing wheel" (km), the mean micro-hardness HV, micro-hardness HV0.05, the relative expanded uncertainty U_{rel} , and the speed of the indenter's penetration v.

Polishing Time (s)	The "Path" of the Sample (km)	Spineline				Diamond			
		HV	HV0.05	U _{rel} (%)	ν (μm s ⁻¹)	HV	HV0.05	U _{rel} (%)	ν (μm s ⁻¹)
0	0	36.20	33.21	50.37	1.97	36.20	33.21	50.37	1.97
300	2.4	31.71	31.84	51.20	2.24	28.53	26.99	65.62	3.63
600	4.8	29.87	30.17	53.92	2.56	27.03	26.91	65.87	3.14
1200	9.6	29.43	29.32	55.45	2.37	26.10	26.60	66.40	3.32
1800	14.4	28.37	28.65	56.72	2.25	28.36	29.63	59.87	3.21
2400	19.2	28.94	29.29	55.52	2.31	26.24	26.14	67.73	2.97
3000	24.0	28.12	27.87	58.46	2.36	26.16	26.45	66.98	2.98
3600	28.8	28.12	28.12	57.85	2.59	26.76	26.44	66.95	3.12

 U_{rel} is the expanded uncertainty (k = 2) of the result of the measurement expressed as a percentage. It is calculated according to the standard ISO 6507-2:2018 [24].

The same operator measured the micro-hardness of selected areas on the metallographic surface of the sample according to the standard ISO 6507-1:2018 [1]. The applied loads P were 0.09807 N, 0.24518 N, 0.49035 N, and 0.9807 N with a load duration of 15 s. The speed of the indenter's penetration was calculated using the method described in [25]. The values of the speed (v, in μ m s⁻¹) are shown in Table 1.

The result of the measurement was a "cluster" of 20 indentations in one area. The mean of the micro-hardness of individual clusters HV, the micro-hardness HV0.05, and its relative expanded uncertainty U_{rel} are shown in Table 1; the values of micro-hardness at individual loads are shown in Figure 1.

Grubbs' test (significance level $\alpha = 0.05$) was used for the detection of statistical outliers. Their presence would indicate a measurement process suffering from special disturbances and out of statistical control. The normality was determined using Freeware Process Capability Calculator software (Anderson–Darling test). The normality and the outliers were determined for files involving values of one "cluster". The values of micro-hardness of all "clusters" have normal distribution without outliers. Their absence suggests the process is unimpeded by gross errors.

Analysis of variance (ANOVA) is a standard statistical technique and can be used to analyze the measurement error and other sources of variability of data in a measurement systems study. The authors used the procedure recommended by Chajdiak and reference manual MSA for calculating the significance of individual factors [26,27]. Two-way ANOVA compares the means of a single variable at different levels of two conditions (factors). A *p* value is used to determine whether a certain pattern they have measured is statistically significant. Statistical significance is a way of saying that the *p* value of a statistical test is small enough to reject the null hypothesis of the test. The most common threshold is p < 0.05, that is, when you would expect to find a test statistic as extreme as the one calculated by your test only 5% of the time. The threshold value for determining statistical significance is also known as the alpha value. According to two-way ANOVA without replication, for spineline, the polishing time (p = 0.000443) and the load (p = 0.000111) both have statistically significant effects on the measured value of the micro-hardness. For diamond paste, the polishing time (p = 0.1896) and the influence of the load (p = 0.0633) are not statistically significant at the significance level $\alpha = 0.05$.



Figure 1. The values of micro-hardness.

The influence of polishing time (s) on the roughness Ra (μ m) measured using a confocal microscope for both axes (x and y perpendicular to each other) is shown in Figure 2. Ra values are presented together for both axes since, by using an unpaired *t*-test, the difference between the Ra values in both axes is not statistically significant (p = 0.9182 for spineline and p = 0.9727 for diamond), and the scratches and grooves on the metallographic surface of the samples do not have a preferred direction. As the polishing time increases, the roughness decreases. But, as can be seen from the picture, the relationship is not completely ideal, especially with longer polishing times. This is evidenced by the values of the Pearson correlation coefficient r and the determination coefficient R^2 (Table 2). A correlation coefficient is a number between -1 and 1 that indicates the strength and direction of a relationship between variables. The coefficient of determination is a number between 0 and 1 that measures how well a statistical model predicts an outcome. Table 2 also shows indices (a—slope and b—intercept) describing the linear dependence. The parameter "b" is the value of y (x = 0) at the intersection of the line with the y-axis at x = 0. If the polishing time exceeds 1800 s, the roughness practically does not decrease. The particles of both polishing agents produce new scratches and grooves, which are partially removed by further polishing; at the same time, additional scratches and grooves are created. At the given granularity of the agent, the process stabilizes after a certain time, and its further continuation makes no sense. We do not state the dependence of roughness on the polishing time, measured with a contact tester. It is even less informative than when measured with a confocal microscope. The roughness values measured in this way are an order of magnitude higher (for example polishing time 1200 s, spineline: 50.0 µm contact tester, and 2.75 µm confocal microscope).

Polishing Agent		Spineli	ne		Diamond			
Indices and Coefficients	a(x)	b	r	R ²	a(x)	b	r	R ²
Figure 2	-0.7348	3.9259	-0.659	0.4341	-0.7182	4.3394	-0.675	0.4559
Figure 3	-0.0502	2.0403	-0.737	0.5439	-0.0491	2.057	-0.758	0.5739
Figure 4	0.073	0.513	-	0.4038	0.0846	0.3681	-	0.8011
Figure 5	0.2127	138.83	-	0.0421	0.1382	129.13	-	0.0159
Figure 6	0.0039	-0.0666	-	0.512	0.003	-0.0566	-	0.1580
Figure 7	0.0005	0.0038	-	0.377	0.0002	0.0041	-	0.2582
Figure 8	-0.0268	2.7249	0.833	0.8449	-0.0181	2.4246	0.624	0.4923
Figure 9	-0.0012	0.0341	-	0.1816	-0.0012	0.0315	-	0.1300

Table 2. Values of indices (a—slope and b—intercept) describing the linear dependence and coefficients of correlation r and determination R^2 .



Figure 2. Relationship between polishing time and roughness Ra, measured with a confocal microscope.



Figure 3. The relationship between Ra and Meyer's index n.



Figure 4. The relationship between Ra and parameter a₁.



Figure 5. The relationship between Ra and parameter a_2 .



Figure 6. The relationship between Ra and c_0 .



Figure 7. The relationship between Ra and a_1/a_2 .



Figure 8. The relationship between micro-hardness HV and Meyer's index n.



Figure 9. The relationship between Ra and W.

The points in the graphs represent measured values or parameter values calculated from measured values. The variance in the experimental points is really high, resulting in a low value (Figures 5 and 9 in Table 2). The chosen linear approximation used in the graphs serves to illustrate the trend.

3. Results

The calculation of Meyer's index and related parameters is detailed in, e.g., Petrík et al., 2023, or Šolc et al., 2023, using [4–6,12,28–30].

Meyer's power law and proportional specimen resistance (PSR) are two principal approaches to describe the ISE quantitatively. The value of Meyer's index n or the work hardening coefficient is n < 2 for "normal" ISE, and n > 2 for reverse ISE. If n = 2, the micro-hardness is independent of the load and is given by Kick's law. The relationship between Ra and Meyer's index n is shown in Figure 3. The value of Meyer's index n decreases with increases in the roughness. The values of the Pearson correlation coefficient r and the determination coefficient R^2 are shown in Table 2.

The nature of the ISE changes from "normal" ISE for ground (unpolished) samples with n = 1.7 to values close to the validity of Kick's law even after a short polishing time. As the polishing time increased, the value of n rose slightly above 2, i.e., into the reverse region (RISE) for samples polished with spineline.

Calculated using two-way ANOVA without replication, for spineline, the polishing time (p = 0.0012738) has a statistically significant effect on the measured value of Meyer's index n but the polishing agent does not (p = 0.786067) at the significance level $\alpha = 0.05$. The fact that the agent does not have a statistically significant influence on the value of Meyer's index was also confirmed by a two-tailed *t*-test (p = 0.8856).

Using a proportional specimen resistance model (PSR), a modified form of the Hays–Kendall approach, yields the parameters a_1 and a_2 . Parameter a_1 (N mm⁻¹) is related to elastic properties and characterizes the load dependence of micro-hardness. It consists of two components: the elastic resistance of the sample and the friction resistance at the indenter facet/sample interface. Parameter a_2 (N mm⁻²) is related to the elastic and plastic properties of the specimen.

The influence of the roughness on the values of parameters a_1 and a_2 can be seen in Figures 4 and 5, respectively. The value of both parameters increases with increasing roughness; the difference is more pronounced for a_1 with a significantly lower correlation for parameter a_2 . The values of the determination coefficient R² are shown in Table 2.

As mentioned in the introduction, the growth of the a_1 parameter with the growth of roughness was also observed by Chuah and Ripin in the analysis of stainless steel, copper, and aluminum samples [16].

Due to the very low values of the coefficient of determination R^2 (Figure 5, Table 2) for the relationship between the roughness Ra and the parameter a_2 , the determination of "true hardness" using a_2 has no meaning.

The parameter c_0 (N) can be calculated using an equation based on a modified form of the PSR model. It is associated with residual surface stress in the sample. The relationship between Ra and parameter c_0 can be seen in Figure 6. For both polishing agents, the residual surface stress increases with the polishing time and thus with the decrease in roughness, and its negative tensile component increases. As it follows from the values of the coefficients of determination R² in Table 2, the dependence is tighter for spineline than for diamond paste.

The ratio a_1/a_2 is a measure of the residual stress due to machining and polishing. As can be seen in Figure 7 and Table 2, residual stress decreases with a decrease in roughness for both polishing agents.

Meyer's index n decreases with increasing average micro-hardness HV, as can be seen in Figure 8. As it follows from Table 2, the values of the coefficients of determination R^2 , the dependence is strong and tighter for spineline than for diamond paste. As the micro-hardness increases, the "normal" character of the ISE, typical for materials with lower

plasticity, increases. Reverse ISE and an inverse relationship between the micro-hardness and n was observed for CRMs made of iron or heat-treated steel with micro-hardness between 195 HV0.05 and 519 HV0.05, heat-treated carbon steel and aluminum alloy EN 6082, or technically pure metals such as Al, Zn, Cu, Fe, Ni, and Co [31,32]. Except for grinding and polishing, the given examples were not deformed.

Hays and Kendall proposed the existence of minimum test load W (N) necessary to initiate plastic deformation; the relationship between the roughness Ra and load W can be seen in Figure 9. As the roughness increases, the load value increases, and the load value varies in the range between 0.0019 and 0.0384 N. As already mentioned above, despite the declared pressure force of the touch tester, traces of the tip of the tester's arm were visible (with a microscope) on the surface of the sample. Thus, plastic deformation occurred, although it was not expected. This anomaly regarding load W was already observed by Petrík et al. and deserves a more detailed analysis [33].

4. Discussion

As for the influence of the polishing time on the quality of the metallographic surface, with the use of both agents, it was possible to achieve a shiny, mirror-like surface when viewed with the naked eye. Even with long polishing times (extremely 3600 s), no affected (Beilby) layer was formed. As the polishing time increases to approx. 1800 s, the roughness of Ra decreases, after which time it stabilizes for both agents without the influence of further polishing. Microscopically, it looks like older scratches and grooves gradually smooth out and disappear. At the same time, due to the inhomogeneity of the agents (larger or harder particles), new scratches are created, which are gradually smoothed out. That is, the process will continue indefinitely, or until over-polishing. The agents used will probably never be able to completely remove the scratches, and extending the polishing time makes no sense. On the contrary, as mentioned below, the polishing time influences the parameters characterizing the ISE and must be taken into account when interpreting them.

As mentioned above by Chuah and Ripin [16], after grinding (no polishing) samples based on stainless steel, copper, and aluminum, an order of magnitude lower roughness Ra ($0.0062-0.1328 \mu m$) was achieved compared to the presented values ($0.87-3.35 \mu m$).

The samples whose ISE-related properties are shown below were polished with spineline for 300 s. If the sample was deformed by tension (tensile test), then with the growth of the local degree of deformation (*Z*), the value of n decreases slightly from RISE (n = 2.1) at zero deformation to slightly "normal" (n = 1.95 at *Z* = 80%). The same course has a dependence between roughness and Meyer's index n. With a decrease in roughness, this goes from significantly "normal" (approx. n = 1.7 in the ground state) to neutral or reverse at lower roughness. Thus, a decrease in roughness has the same effect on Meyer's index as a decrease in the degree of tensile deformation. On the contrary, during deformation by compression, the value of Meyer's index increases from a slightly "normal" to the reverse region (approx. n = 2.2 at ε = 80%) as the degree of deformation increases.

An increase in polishing time, a decrease in roughness, and an increase in the degree of deformation move the values of Meyer's index into the reversal region, characteristic of plastic materials.

For all three materials—stainless steel, copper, and aluminum alloy—the same course of the effect of roughness on Meyer's index was analyzed: with a decrease in roughness, it goes from significantly "normal" to neutral to reverse at lower roughness as stated by Chuah and Ripin [16], Table 1, which corresponds to the results of the authors of this paper (n = 1.72 to 1.98). The most significant changes were observed in the case of stainless steel.

Parameter a_1 characterizes the load dependence of micro-hardness. Its value decreases with increasing polishing time (and thus with decreasing roughness). Thus, for less rough (highly polished) samples, a smaller influence of load on micro-hardness values is expected. As the degree of deformation increases, its value decreases (positive for flat and negative for compressive deformation). The same decrease in the value of coefficient a_1 with a decrease in roughness was also noted by [16,28,33].

Parameter c_0 is associated with residual surface stress in the sample. With the polishing time, the values increase slightly, from zero to negative values at minimum roughness. Thus, with a decrease in roughness, surface tensions increase. As the deformation increases, the (negative) value of c_0 grows, both in compression and tension (more pronounced in the latter).

The ratio a_1/a_2 is the measure of the residual stress due to machining and polishing. Its value decreases with increasing polishing time (and thus with decreasing roughness). As the degree of tensile deformation increases, it rises from negative to positive values; with compression deformation, the trend is the opposite. If we take into account the values of a_1 and a_2 given by Chuah and Ripin for aluminum alloy and calculate the parameter a_1/a_2 from them, then this has the same dependence on roughness—with a decrease in roughness, the value of the parameter and therefore the residual stresses decrease [16].

The longer the sample is polished and the smaller the roughness, the smaller the values of c_0 (residual stresses) and a_1/a_2 (stresses stress due to machining and polishing). It is possible that these stresses induced by grinding and polishing are removed. So, the longer the sample is polished, the smaller they are.

5. Conclusions

- The polishing time has a statistically significant effect on the size of the Meyer's index n, but the polishing agent does not.
- 2. If diamond paste is used as a polishing agent, the resulting micro-hardness is lower than when using spineline.
- 3. There is a correlation between the polishing time (and roughness Ra) and Meyer's index n; with a decrease in roughness, the value of n increases (from "normal" to the reverse character).
- 4. Extending the polishing time above 1800 s with the agents used is not important, as it cannot completely remove scratches.
- 5. When interpreting the parameters characterizing the ISE, it is necessary to take into account the polishing time and subsequent roughness.

Author Contributions: Quality, preparation of samples, J.P., D.D., M.Ť. and S.K.; ISE calculation, P.B. and J.P.; micro-hardness measurement, J.P. and P.B.; translation, statistics, J.P.; literature review, M.Š., M.M. and J.P. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by the Cultural and Educational Grant Agency of the Ministry of Education of the Slovak Republic, KEGA project 009TUKE-4/2023.

Data Availability Statement: Data are contained within the article.

Conflicts of Interest: The authors declare no conflict of interest.

References

- ISO 6507-1; Metallic Materials—Vickers Hardness Test Part 1—Test Method. International Organization for Standardization (ISO): Geneva, Switzerland, 2018.
- ISO 14577-1; Metallic Materials—Instrumented Indentation Test for Hardness and Materials Parameters Part 1—Test Method. International Organization for Standardization (ISO): Geneva, Switzerland, 2015.
- Voyiadjis, G.Z.; Peters, R. Size Effects in Nanoindentation: An Experimental and Analytical Study. Acta Mech. 2010, 211, 131–153. [CrossRef]
- 4. Gong, J.; Wu, J.; Guan, Z. Examination of the Indentation Size Effect in Low-Load Vickers Hardness Testing of Ceramics. *J. Eur. Ceram. Soc.* **1999**, *19*, 2625–2631. [CrossRef]
- 5. Sangwal, K.; Surowska, B.; Błaziak, P. Analysis of the Indentation Size Effect in the Microhardness Measurement of Some Cobalt-Based Alloys. *Mater. Chem. Phys.* 2003, 77, 511–520. [CrossRef]
- Ren, X.J.; Hooper, R.M.; Griffiths, C.; Henshall, J.L. Indentation Size Effect in Ceramics: Correlation with H/E. J. Mater. Sci. Lett. 2003, 22, 1105–1106. [CrossRef]
- Sangwal, K. On the Reverse Indentation Size Effect and Microhardness Measurement of Solids. *Mater. Chem. Phys.* 2000, 63, 145–152. [CrossRef]

- Hakamada, M.; Nakamoto, Y.; Matsumoto, H.; Iwasaki, H.; Chen, Y.; Kusuda, H.; Mabuchi, M. Relationship between Hardness and Grain Size in Electrodeposited Copper Films. *Mater. Sci. Eng. A* 2007, 457, 120–126. [CrossRef]
- Şahin, O.; Uzun, O.; Kölemen, U.; Uçar, N. Mechanical Characterization for β-Sn Single Crystals Using Nanoindentation Tests. Mater. Charact. 2008, 59, 427–434. [CrossRef]
- 10. Navrátil, V.; Novotná, J. Some problems of microhardness of metals. J. Appl. Math. 2009, 2, 241–244.
- Cepova, L.; Kovacikova, A.; Cep, R.; Klaput, P.; Mizera, O. Measurement System Analyses—Gauge Repeatability and Reproducibility Methods. *Meas. Sci. Rev.* 2018, 18, 20–27. [CrossRef]
- Kim, J.-Y.; Kang, S.-K.; Lee, J.-J.; Jang, J.; Lee, Y.-H.; Kwon, D. Influence of Surface-Roughness on Indentation Size Effect. Acta Mater. 2007, 55, 3555–3562. [CrossRef]
- 13. Kim, J.-Y.; Kang, S.-K.; Greer, J.R.; Kwon, D. Evaluating Plastic Flow Properties by Characterizing Indentation Size Effect Using a Sharp Indenter. *Acta Mater.* 2008, *56*, 3338–3343. [CrossRef]
- 14. Xia, Y.; Bigerelle, M.; Marteau, J.; Mazeran, P.; Bouvier, S.; Iost, A. Effect of Surface Roughness in the Determination of the Mechanical Properties of Material Using Nanoindentation Test. *Scanning* **2014**, *36*, 134–149. [CrossRef] [PubMed]
- Alsoufi, M.S.; Alhazmi, M.W.; Ghulman, H.A.; Munshi, S.M.; Azam, S. Surface Roughness and Knoop Indentation Micro-Hardness Behavior of Aluminium Oxide (Al₂O₃) and Polystyrene (C8H8)n Materials. *Int. J. Mech. Mechatron. Eng.* 2016, 16, 43–49.
- Chuah, H.G.; Ripin, Z.M. Quantifying the Surface Roughness Effect in Microindentation Using a Proportional Specimen Resistance Model. J. Mater. Sci. 2013, 48, 6293–6306. [CrossRef]
- Erdemir, U.; Sancakli, H.S.; Yildiz, E. The Effect of One-Step and Multi-Step Polishing Systems on the Surface Roughness and Microhardness of Novel Resin Composites. *Eur. J. Dent.* 2012, *06*, 198–205. [CrossRef]
- 18. Korkmaz, Y.; Ozel, E.; Attar, N.; Aksoy, G. The Influence of One-Step Polishing Systems on the Surface Roughness and Microhardness of Nanocomposites. *Oper. Dent.* **2008**, *33*, 44–50. [CrossRef] [PubMed]
- ISO 6892-1; Metallic Materials—Tensile Testing. Part 1: Method of Test at Room Temperature. International Organization for Standardization (ISO): Geneva, Switzerland, 2016.
- Cuff, T. Beilby Layer. 2016. Available online: https://www.researchgate.net/publication/297761245_Beilby_Layer?channel=doi& linkId=56e338f508ae98445c1b2d31&showFulltext=true (accessed on 30 September 2023).
- 21. ISO 4287-1; Surface Roughness—Terminology—Part 1: Surface and Its Parameters. International Organization for Standardization ISO: Geneva, Switzerland, 1997.
- Mitutoyo Surftest-SJ-310. Available online: https://shop.mitutoyo.eu/web/mitutoyo/en/mitutoyo/1292249267209/Surftest%20 SJ-310%20%5Bmm%5D/\$catalogue/mitutoyoData/PR/178-570-01D/index.xhtml (accessed on 2 September 2023).
- 23. *ISO 6507-2;* Metallic Materials—Vickers Hardness Test Part 2—Verification and Calibration of Testing Machines. International Organization for Standardization (ISO): Geneva, Switzerland, 2005.
- ISO 6507-2; Metallic Materials-Vickers Hardness Test—Verification and Calibration of Testing Machines. International Organization for Standardization (ISO): Geneva, Switzerland, 2018.
- Petrík, J.; Palfy, P.; Blaško, P.; Girmanová, L.; Havlík, M. The Indentation Size Effect (ISE) and the Speed of the Indenter Penetration into Test Piece. *Manuf. Technol.* 2016, 16, 771–777. [CrossRef]
- 26. Chajdiak, J. Štatistika v Exceli 2007, 2nd ed.; STATIS: Bratislava, Slovakia, 2009.
- 27. Chrysler Group. Measurement Systems Analysis: Reference Manual, 4th ed.; Chrysler Group: Detroit, MI, USA, 2010; ISBN 978-1-60534-211-5.
- 28. Petrík, J.; Blaško, P.; Markulík, Š.; Šolc, M.; Palfy, P. The Indentation Size Effect (ISE) of Metals. Crystals 2022, 12, 795. [CrossRef]
- 29. Kim, H.; Kim, T. Measurement of Hardness on Traditional Ceramics. J. Eur. Ceram. Soc. 2002, 22, 1437–1445. [CrossRef]
- Li, N.; Liu, L.; Zhang, M. The Role of Friction to the Indentation Size Effect in Amorphous and Crystallized Pd-Based Alloy. J. Mater. Sci. 2009, 44, 3072–3076. [CrossRef]
- Karaca, I.; Büyükakkas, S. Microhardness Characterization of Fe- and Co-Based Superalloys. Iran. J. Sci. Technol. Trans. Sci. 2019, 43, 1311–1319. [CrossRef]
- Cai, X.; Yang, X.; Zhou, P. Dependence of Vickers Microhardness on Applied Load in Indium. J. Mater. Sci. Lett. 1997, 16, 741–742. [CrossRef]
- 33. Petrík, J.; Blaško, P.; Petryshynets, I.; Mihaliková, M.; Pribulová, A.; Futáš, P. The Influence of the Degree of Tension and Compression of Aluminum on the Indentation Size Effect (ISE). *Metals* **2022**, *12*, 2063. [CrossRef]

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