PD.TVZ.HR
POLYTECHNIC & DESIGN Vol. 12, No. 4, 2024.
Mrežna inačica: ISSN 2459-6302
Tiskana inačica: ISSN 1849-1995
DOI: 10.19279/TVZ.PD.2024-12-4-14

IZRAČUN MEHANIČKOG TROŠENJA MATERIJALA PRILIKOM PRIPREME UZORAKA ZA METALOGRAFSKU ANALIZU

CALCULATION OF MECHANICAL WEAR OF MATERIAL DURING SAMPLE PREPARATION FOR METALLOGRAPHIC ANALYSIS

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SAŽETAK

Uzorci se za metalografsku analizu, odnosno analizu mikrostrukture materijala, pripremaju preciznim brušenjem i poliranjem. Prilikom pripreme uzoraka za metalografsku analizu javlja se mehaničko trošenje materijala. U ovom radu od interesa je bilo saznati, odnosno izračunati, stopu trošenja tri različita materijala u pojedinim koracima pripreme uzoraka brušenjem i poliranjem, kao i isto dovesti u korelaciju s vrstom materijala. Za izračun su odabrana tri različita uzorka koja su istovremeno i pod istim uvjetima podvrgnuta preciznom brušenju u četiri perioda, odnosno više koraka na različitim granulacijama abrazivne podloge. Nakon određenih vremenskih perioda, na svim uzorcima provedena su mjerenja u dvadeset i osam točaka. Ideja ovog rada bila je izrada kalkulacije za lakšu, vremenski precizniju i sigurniju pripremu uzoraka čime se poboljšava učinkovitost osobe koja priprema uzorak, smanjuje se rizik uništavanja uzorka i smanjuje se ljudski stres prilikom obavljanja rada.

Ključne riječi: *Metalografska ispitivanja, mikrostruktura materijala, brušenje i poliranje materijala, Prestonov koeficijent*

ABSTRACT

Samples for metallographic examination, i.e., examination of the microstructure of the material, are prepared by precise grinding and polishing. "During the preparation of samples for metallographic examination, mechanical wear of the material occurs. This study aims to determine, and calculate, the wear of three different materials at individual steps of the sample preparation through grinding and polishing, as well as its correlation with the type of material. For the calculation, three different samples were selected, which were simultaneously subjected under the same conditions of precision grinding in four periods, i.e., several steps on different grits of the abrasive substrate. After certain time periods, measurements were conducted at twenty-eight points on all four samples. The idea of this study was to create a calculation for a more efficient, time-precise, and safer sample preparation process, thereby improving the effectiveness of the person preparing the sample, reducing the risk of sample destruction, and minimizing human stress during the task.

Keywords: *Metallography, microstructure, material grinding and polishing, Preston's coefficient*

1. UVOD
1. INTRODUCTION

Metallographic analysis is the analysis of the microstructure of metals, from which the properties and any irregularities of the analysed samples can be determined. The actual "reading" of the microstructure is preceded by steps such as selecting the metal, precise cutting, mounting, surface preparation by grinding and polishing, as well as etching with special solutions for a certain period of time depending on the type of material. Microstructure analysis

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can also provide insights into the processes to which the tested material has been subjected, for example, whether normalization has been performed, whether the material has been surface or through-hardened or annealed, and whether it has been processed by rolling or some other technological deformation process. Through microscopic analysis, it is possible to determine the size and number of crystal grains on the surface of the observed material, which also allows for the identification of any changes in the microstructure. Additionally, different metals can have significantly different microstructures and present phases (crystals, intermetallic compounds, inclusions, nodules), so metallographic analysis can also be used for material identification.[1]

In this paper, the focus was to determine and calculate the material loss (of steel and aluminium) during the individual steps of sample preparation by grinding and polishing, and to correlate this with the type of material. This can be particularly important for thin materials such as sheets, where the sample can be destroyed during preparation.

2. PRIPREMA UZORAKA ZA METALOGRAFSKU ANALIZU

2. SAMPLE PREPARATION FOR METALLOGRAPHIC ANALYSIS

Before preparing samples for metallographic analysis, a piece according to dimensions that fit the mould has to be cut first. The sample is cut using precision cutting machines that allow sample cooling during the process to prevent microstructural changes caused by sample heating. Additionally, precision cutting machines enable obtaining low-roughness surfaces after cutting, which simplifies and shortens subsequent sample preparation steps for analysis. After cutting, the process continues with mounting, several grinding and polishing steps, and finally sample etching. During cutting, it is crucial to focus on obtaining a representative sample that will provide the most information about potential irregularities in the material. After cutting, the surface to be placed at the bottom of the mould is selected and marked – this surface will be analysed in the subsequent procedure. After placing the metal test piece into the mould, resin is prepared and poured into the mould to fill the empty space around the metal. [1]

The resin that meets the needs of preparation and analysis, that is, the condition that it wears at the same rate as the metal, is obtained by mixing three components:

1. Base liquid component,
2. Powdered component for achieving strength,
3. Liquid component for hardening the mixture.

Hardening occurs only after the chemical reaction that results from mixing all three components. The hardening process begins immediately, but it takes a longer time to reach full hardness. The curing time of the resin, until the next stage of sample preparation can begin, lasts at least 12 hours. All components create a type of resin that is resistant to wear, which means that during grinding, the resin does not wear faster than the tested metal sample and is also resistant to the effects of acids and other etching agents. An important note is that during “mounting,” chemicals are used that can be harmful to health, both by contact and by inhaling their vapours, so it is mandatory to use protective gloves and safety glasses and, if possible, a fume hood.

After the resin has fully hardened, the second step of sample preparation follows, consisting of grinding and polishing the test surface of the sample. The grinding process consists of several steps and is determined by the type of metal being processed and its characteristics. In specialized instructions for sample preparation (manufacturer Struers, instructions supplied with the Labo-Pol 5 machine), for each step, the grit size of the abrasive paper or the type of grinding plate is specified, as well as the duration of the procedure (Figure 1). In addition to the processing time and the type of abrasive paper or plate, the number of revolutions of the grinding or polishing surface and the pressure applied to the sample during grinding are also specified. The guidelines for sample preparation also specify the type of abrasive suspension for individual steps, which for high-alloy steels contains abrasive granules of 3–9 μm in size.

Brušenje

Korak		
Površina	MD-Piano 220	MD-Allegro
Vrsta Abrazivne površine		DiaPro AllegroLargo 9
Granulacija		9 µm
Suspenzija		
Broj okretaja	300	150
Sila opterećenja	40	30
Vrijeme		3

Poliranje

Korak	DP	OP
Površina	MD-Dac	MD-Chem
Vrsta Abrazivne površine	DiaPro Dac 3	OP-A
Granulacija	3 µm	
Suspenzija		
Broj okretaja	150	150
Sila opterećenja	30	15
Vrijeme	3	2

Slika 1 *Koraci i sredstva za brušenje i poliranje iz Struers priručnika*

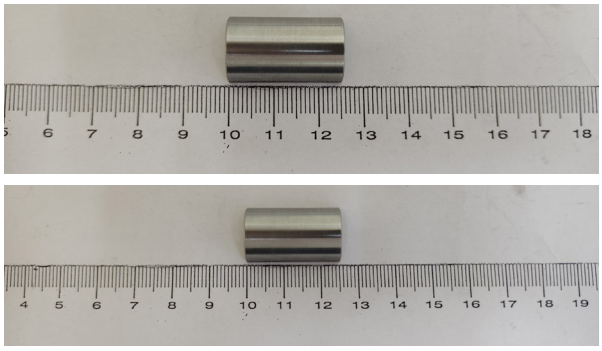
Figure 1 *Steps and means for grinding and polishing from the Struers guide*

During the process of grinding and polishing samples, attention must be paid to the condition of the test surface to avoid unnecessary material loss and to determine the optimal moment to change the grit level. If the process switches to a finer step prematurely, the procedure will result in significantly greater time consumption and material usage for grinding or polishing. The grit size of the suspension used in the final polishing step is up to 3 µm.

After polishing is completed, the samples undergo etching according to precisely defined recipes depending on the type of alloy. Metal etching agents primarily include chloridic and sulfuric acids of varying concentrations, followed by phosphoric, hydrofluoric, nitric, and other acids. To prevent excessive dissolution of the metal surface, protective agents (inhibitors) are added to the etching baths.

For this study, three different samples of varying hardness were selected. Hardness is an inversely proportional factor during grinding, meaning higher hardness results in slower and lesser wear (material loss). All three samples have the same diameter. The first sample is made of pure mounting resin, the second sample is aluminium mounted in the same resin, and the third sample is low-alloy steel, also embedded in the same resin.

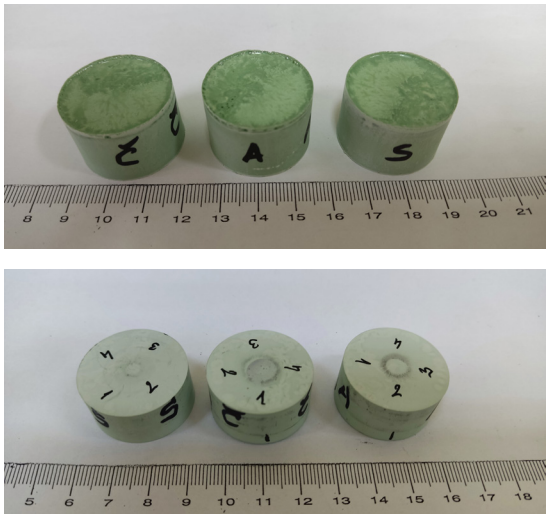
When selecting materials (steel and aluminium), samples with the same cross-sectional area of 9 mm² were chosen (Figure 2). Given the assumption that materials of different hardness may wear differently, it was essential to maintain identical grinding parameters, such as the rotational speed of the polishing machine head, the rotational speed of the abrasive medium's base, the applied pressure on the sample, etc.



Slika 2 *Uzorci metala površine poprečnog presjeka 9 mm²*

Figure 2 *Metal samples with a cross-sectional area of 9 mm²*

For all samples, the same resin was used as is commonly applied in practice for the preparation of metal samples for various metallographic and optical examinations as well as hardness testing. In order to perform the measurements in a specific sequence and to avoid errors, lines (symmetry axes) were marked on the samples, along which, after each grinding step, measurements were carried out in the same order (Figure 3b).



a)

b)

Slika 3 *a) Fotografija ukalupljenih uzoraka, b) Fotografija uzoraka s označenim simetralama*

Figure 3 *a) Photo of moulded samples, b) Photo of samples with marked bisectors*

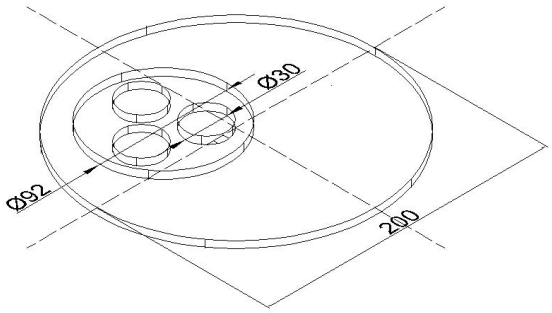
After embedding the samples in resin, the first step of the examination was to take measurements of each sample as the initial reference dimension, where the designation "S" indicates the measured symmetry axis (Table 1).

Tablica 1 *Početne mjere debljine uzoraka*

Table 1 *Starting sample hight measurements*

S1	S2	S3	S4
14.297	14.422	14.461	14.332
14.385	14.524	14.494	14.394
14.491	14.529	14.519	14.439
14.520	14.56	14.553	14.484
14.575	14.562	14.561	14.511
14.576	14.659	14.593	14.560
14.608	14.615	14.622	14.612
Srednja vrijednost			
14.493	**14.553**	**14.543**	**14.476**

Grinding and polishing were performed on the "Struers LaboPol-5" device. This is an automatic polishing device that accommodates samples with a diameter of 30 mm or 40 mm (Figure 4).



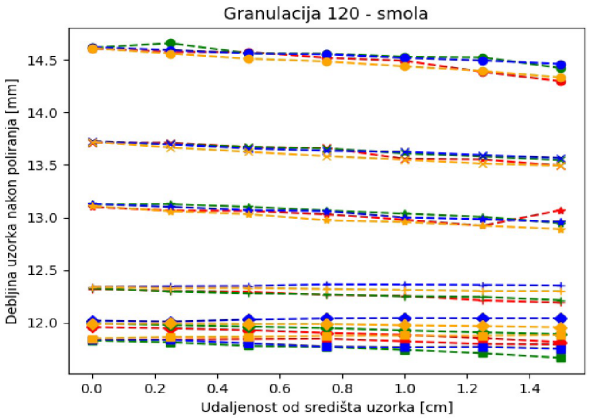
Slika 4 *Položaj uzoraka na uređaju*

Figure 4 *Sample placement on the machine*

3. IZRAČUN TROŠENJA MATERIJALA PRILIKOM PRIPREME UZORAKA ZA METALOGRAFSKA ISPITIVANJA

3. CALCULATION OF MATERIAL WEAR DURING THE SAMPLE PREPARATION FOR METALLOGRAPHIC EXAMINATION

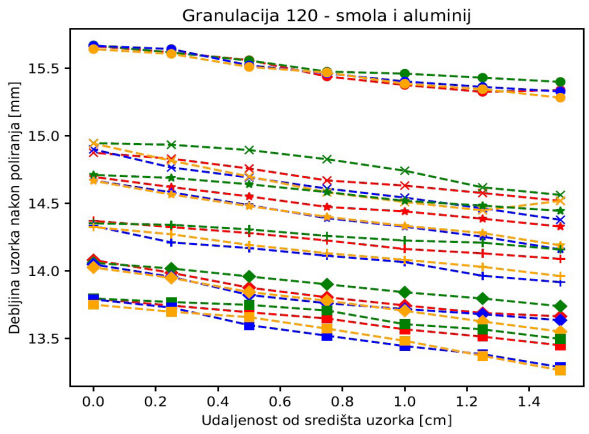
When discussing wear calculation, the primary emphasis was placed on the required sample preparation time and sample thickness. Calculating the time increases laboratory efficiency and reduces the risk of errors. Regarding sample thickness, especially for thin samples, it is crucial to control the time to prevent excessive grinding and consequent "disappearance" of the sample. The material wear calculation shown in the analysis (Figures 5-7) refers to the 120-grit abrasive paper. As previously stated, since three different materials or material combinations were used, different wear values were obtained. The resin used for embedding the materials has a hardness value approximating that of the metal, with the key factor for wear rate being the type of metal embedded in the resin. The results showed that wear rate depends linearly on the type and hardness of the metal. Among the three tested samples, the fastest wear was measured in the resin, followed by the resin-aluminium combination, and finally, the slowest wear was exhibited by the resin-steel combination.



Slika 5 *Grafički prikaz trošenja smole*

Figure 5 *Graphic representation of resin sample wear*

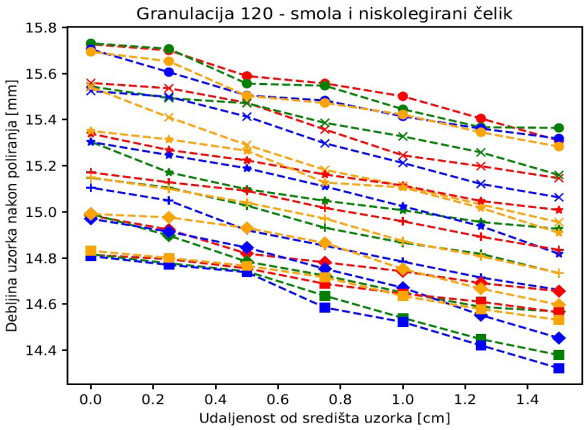
From the presented graph (Figure 5), it is evident that the material (resin) is homogeneous and uniform across the entire diameter, and there are very small deviations in wear between the edge and the centre of the sample.



Slika 6 *Grafički prikaz trošenja smole i aluminija*

Figure 6 *Graphic representation of resin and aluminium wear*

The graph (Figure 6) shows the abrasion or wear of the material (resin and aluminium) during grinding, i.e., the graph indicates that the sample wears more at the edges due to the difference in hardness between the aluminium and the resin.



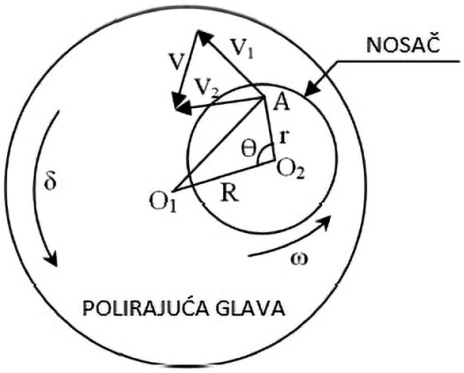
Slika 7 *Grafički prikaz trošenja smole i čelika*

Figure 7 *Graphic display of resin and steel wear*

The differences in wear between the edge area and the centre closer to the steel embedded in the resin are by far the greatest (Figure 7), because the difference in hardness between these two materials is the largest. This finding confirms, as previously mentioned, that wear is linearly dependent on the hardness of the material. This phenomenon leads to the formation of convexity in the sample, making it more difficult to observe the microstructure.

The sample is attached to a stainless-steel holder with a thickness of 2 mm, a diameter of 46 mm, and the capacity to hold 3 samples simultaneously, whose central symmetry axes are mutually rotated by 120°. The pressure of the samples against the abrasive surface (F) is constant at 30 N, the rotational speed of the holder plate head (ω) is 250 rpm, and the rotational speed of the plate (δ) is 250 rpm. The distance from the centre of the sample O_2 to point A on the holder plate is r = 27 mm, and the distance from the centre of the polishing plate O_1 to the centre of the holder plate O_2 is R = 42 mm. Below is a simplified diagram of the polishing movement (Figure 8), which includes the rotation of the polishing plate and the rotation of the head. The velocity at point A on the polishing plate relative to O_1 is V_1, and the velocity at point A on the sample relative to O_2 is V_2, so the relative velocity of the sample and the polishing plate at point A (V) is given by expression (1):

$$V(r,\theta) = [R^2\delta^2 + r^2(\delta-\omega)^2 + 2rR\delta(\delta-\omega)\cos\,\theta]^{1/2} \quad (1)$$



Slika 8 *Pojednostavljeni prikaz gibanja polirajuće glave i diska*

Figure 8 *Simplified view of polishing head and carrier disc*

The relationship between material removal rate and polishing velocity V, applied pressure P, and other external factors, as shown in equation (2) [3]:

$$\frac{dh}{dt} = kPV = kP\frac{ds}{dt} \quad (2)$$

The proportionality coefficient, also known as the "Preston coefficient" k, is determined experimentally and depends on process parameters (material, abrasion, lubrication, etc.). The contact between the sample and the disk changes dynamically over time. This can cause additional uncertainties in polishing research, so three main assumptions have been made [4]:

1. The sample and polishing disk remain in complete contact without separation,
2. The applied pressure does not change during the polishing time (this does not mean that the pressure distribution must be constant everywhere on the contact surface),
3. The proportionality constant k does not change during the polishing time.

The applied pressure does not need to be uniformly distributed across the entire contact surface but can vary with respect to the distance from the centre of the sample base r, the angle θ, or over time t. By integrating the previous

first-order differential equation, we obtain the following expression [3]:

$$h(r) = k \int_0^T P(r,t) \cdot V(r,t) dt \tag{3}$$

By substituting expression (1) into expression (3):

$$h(r) = k \int_0^T P(r,t) \cdot [R^2\delta^2 + r^2(\delta - \omega)^2 + 2rR\delta(\delta - \omega)\cos\ \theta]^{1/2} dt \tag{4}$$

It is important to note that in the previous expressions, the pressure distribution *P* on the contact surface between the sample and polishing disk is a function of distance and time: *P = P(r,t)*. The indentation into the abrasive plate, i.e., the indentation depth d due to the normal (applied) force *F* acting on a rigid cylindrical sample with a base radius *R* (Figure 9), is given by the expression:

$$d = \frac{F}{2\ R\ E^*} \tag{5}$$

Where the effective *Young's* elasticity modulus of the sample and the abrasive plate in contact is defined as:

$$\frac{1}{E^*} = \frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2} \tag{6}$$

Young's modulus of the material is given with E_1 and E_2, while Poisson's ratios ν_1 and ν_2.

$$F = 2\ R\ E^* d \tag{7}$$

Pressure distribution on the contact surface of the rigid cylindrical sample is given with the expression

$$p(r) = p_0 \left(1 - \frac{r^2}{R^2}\right)^{-1/2} \tag{8}$$

where *r* represents the distance between the point on the contact surface and the centre of the sample base. The applied force *F* corresponds to the integral of the pressure distribution over the surface element:

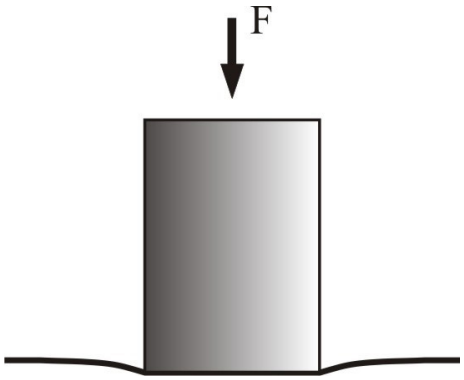
$$F = \int_0^R \int_0^{2\pi} p(r)\ r\ dr\ d\theta = p_0 \int_0^R \int_0^{2\pi} \left(1 - \frac{r^2}{R^2}\right)^{-1/2} r\ dr\ d\theta = 2\pi R\ |R|\ p_0 \tag{9}$$

Clearly, it must hold that $2\pi R\ |R|\ p_0 = 2\ R\ E^*\ d$ i.e., the constant p_0 is:

$$p_0 = \frac{1}{\pi} E^* \frac{d}{R} \tag{10}$$

The pressure distribution from expression (8) proved to be unrealistic at the edges of the resin sample, so in the case of linear regression, the decision is made for the case of a constant amount of pressure of the rigid cylindrical (roller) indenter on the substrate, i.e. (Figure 9):

$$p(r) = p_0 \tag{11}$$



Slika 9 *Kontakt između krutog cilindričnog (valjkastog) utiskivača i elastičnog poluprostora*

Figure 9 *Contact between a rigid cylindrical identor and elastic half-space*

4. ODREĐIVANJE PRESTONOVOG KOEFICIJENTA

4. DETERMINATION OF PRESTON'S COEFFICIENT

The pressure distribution from expression (8) has proven unrealistic at the edges of the resin sample, so in the case of linear regression, the scenario of a constant pressure magnitude for a rigid cylindrical (rod-shaped) indenter on the substrate is chosen:

$$h(t) = k \cdot p_0 \cdot R \cdot \delta \cdot t \quad (12)$$

i.e.

$$h(t) = a \cdot t + b \quad (13)$$

During the measurement, $\omega = \delta = 250$ rpm ≈4.167 rps were set, and $R = 4.2$ cm was measured. For the resin case, linear regression yields the values of the slope coefficient a and intercept b that best fit the measurements for all 4 measurement lines, as shown (Table 1). From the slope coefficient a, the "Preston" coefficient k can be easily calculated using the expression:

$$k = \frac{a}{p_0 \cdot R \cdot \delta} \quad (14)$$

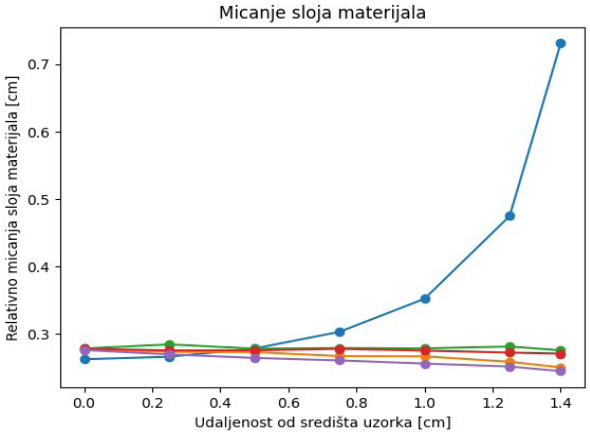
Since the value didn't change, the result of the fraction for all of these cases is 74,28 N/(s·cm). The value of the averaged Preston's coefficients for each measuring line on the sample are also shown (Table 2).

Tablica 2 *Koeficijenti smjera i odsječka pravca kod linearne regresije dobiveni pomoću vlastitog koda, te Prestonovi koeficijenti za sve 4 mjerne linije na uzorku pri korištenju granulacije 120.*

Table 2 *Linear regression direction and line section coefficients obtained using own code, and Preston coefficients for all 4 measuring lines on the sample using grain size 120.*

Measuring line	$\bar{a}\ \left[\frac{\text{mm}}{\text{s}}\right]$	$\bar{b}$ [mm]	$p_0\ \left[\frac{\text{N}}{\text{cm}^2}\right]$	$\bar{k}\ \left[\frac{\text{mm}^2}{\text{N}}\right]$
S_1	0.0092	0.2598	4.244	0.1239
S_2	0.0095	0.2641	4.244	0.1279
S_3	0.0092	0.2647	4.244	0.1239
S_4	0.0088	0.2796	4.244	0.1185

Simulations of polishing using the pressure distribution $p(r)=p_0\ (1-(r/R)^2)^{(-1/2)}$ indicate that the relative wear of the material layer agrees well with experimental results for distances r < 0.5 cm from the centre of the sample base. At the edges of the sample, the simulations suggest that the removed layer should be approximately 0.7 cm, which is several times larger than the experimentally obtained values. Therefore, it is considered that this pressure distribution is not applicable in simulations of material layer removal for the polishing device used. The graphical representation of this case is shown below (Figure 10).



Slika 10 *Relativno micanje sloja materijala kao funkcija udaljenosti od središta. Plavom bojom prikazani su rezultati simulacije, dok su zelenom, crvenom, narančastom i ljubičastom debljine maknutog sloja koje odgovaraju različitim linijama na uzorku duž kojih su izvršena mjerenja.*

Figure 10 *The relative displacement of a layer of material as a function of distance from the centre. The blue colours show the simulation results, while green, red, orange and purple are the thicknesses of the removed layer that correspond to the different lines on the sample along which the measurements were made.*

5. DISKUSIJA
5. DISCUSSION

Proper surface preparation of metals through precise cutting, grinding, and polishing procedures is crucial for accurate metallographic analysis. A calculation such as the one presented in this paper can be used to determine the time required for sample preparation. In addition to time, it is also important to know how much material is removed at each grinding step, especially when dealing with samples of low hardness and very small thickness. Sometimes it is necessary to prepare material, for example copper or a copper alloy with high ductility but low hardness, and if grinding is performed for too long, the sample can be destroyed. The idea of this work was to develop a calculation for easier, more time-precise, and safer sample preparation, thereby improving the efficiency of the person preparing the sample, reducing the risk of sample destruction, and reducing human stress during the work.

In future research, based on the measured values of relative material removal, the shape of the pressure distribution should be determined, since there are differences between devices and the available distributions are not applicable in this case.

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6. REFERENCES

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• **Deni Vale** - A lecturer in the field of physics completed the integrated undergraduate and graduate study of physics – research track at the Faculty of Science (PMF), University of Zagreb, and earned the academic title of Master of Physics. He worked for 5 years as an assistant at the Department of Theoretical Physics, Division of Physics, PMF in Zagreb, as well as a teacher of physics and mathematics in several primary and secondary schools in the Istria County. In 2023, he was appointed to the teaching title and position of lecturer at the Istrian University of Applied Sciences (IV) in Pula. At IV, he is the course leader for the following subjects: Embedded Computer Systems Design, Applied Mathematics, Physics, Dynamic Systems Modelling, and Neural Networks. He is currently pursuing a doctoral degree within the postgraduate doctoral program at the Faculty of Science, University of Zagreb, with the topic: “Collective Nuclear Excitations in the Relativistic Second Random Phase Approximation” under the supervision of Dr. Nils Paar.

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