

Tailoring MAO Coatings on Pure Magnesium via Electrical Parameters in a Multi-Component Electrolyte

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INTRODUCTION & AIM

Micro-arc oxidation (MAO) is an advanced surface modification technique based on a high-voltage electrochemical process that generates plasma micro-discharges on a metal surface. As a result, hard, porous ceramic oxide coatings with excellent adhesion to the substrate are formed. By adjusting processing parameters, MAO enables control over coating morphology, thickness, composition, and functional properties, while also allowing the incorporation of electrolyte-derived elements into the growing oxide coating [1].

Magnesium is a particularly attractive material for MAO treatment due to its potential use in biodegradable orthopedic implants. Its high biocompatibility and elastic modulus, which is closer to that of natural bone than conventional metallic implant materials, make it a promising candidate for biomedical applications. However, the rapid degradation rate and relatively poor mechanical properties of pure magnesium limit its direct clinical use, highlighting the need for effective surface engineering strategies [2, 3].

The aim of this study was to investigate the influence of MAO electrical parameters, particularly applied voltage and current, on the formation and properties of coatings produced on pure magnesium in a Ca/P/Si/Cu-containing electrolyte. Special attention was paid to coating morphology, elemental incorporation, topography, thickness, mechanical performance, adhesion, wettability, and surface free energy. The ultimate goal was to identify processing conditions that enable tailoring of coating characteristics toward biomedical applications.

METHODOLOGY

High-purity magnesium cylindrical specimens were ground, ultrasonically cleaned, and subjected to MAO treatment in a multi-component electrolyte containing Ca, P, Si, and Cu species. The processing parameters are summarized in Table 1 and the MAO setup is illustrated in Figure 1.

Table 1. Electrical parameters applied during the MAO process and specimen designation.

Current	Current density	Voltage	Specimen designation
180	22.8 mA/cm ²	300, 350 or 400 V	180_300, 180_350, 180_400
280	35.4 mA/cm ²	300, 350 or 400 V	280_300, 280_350, 280_400

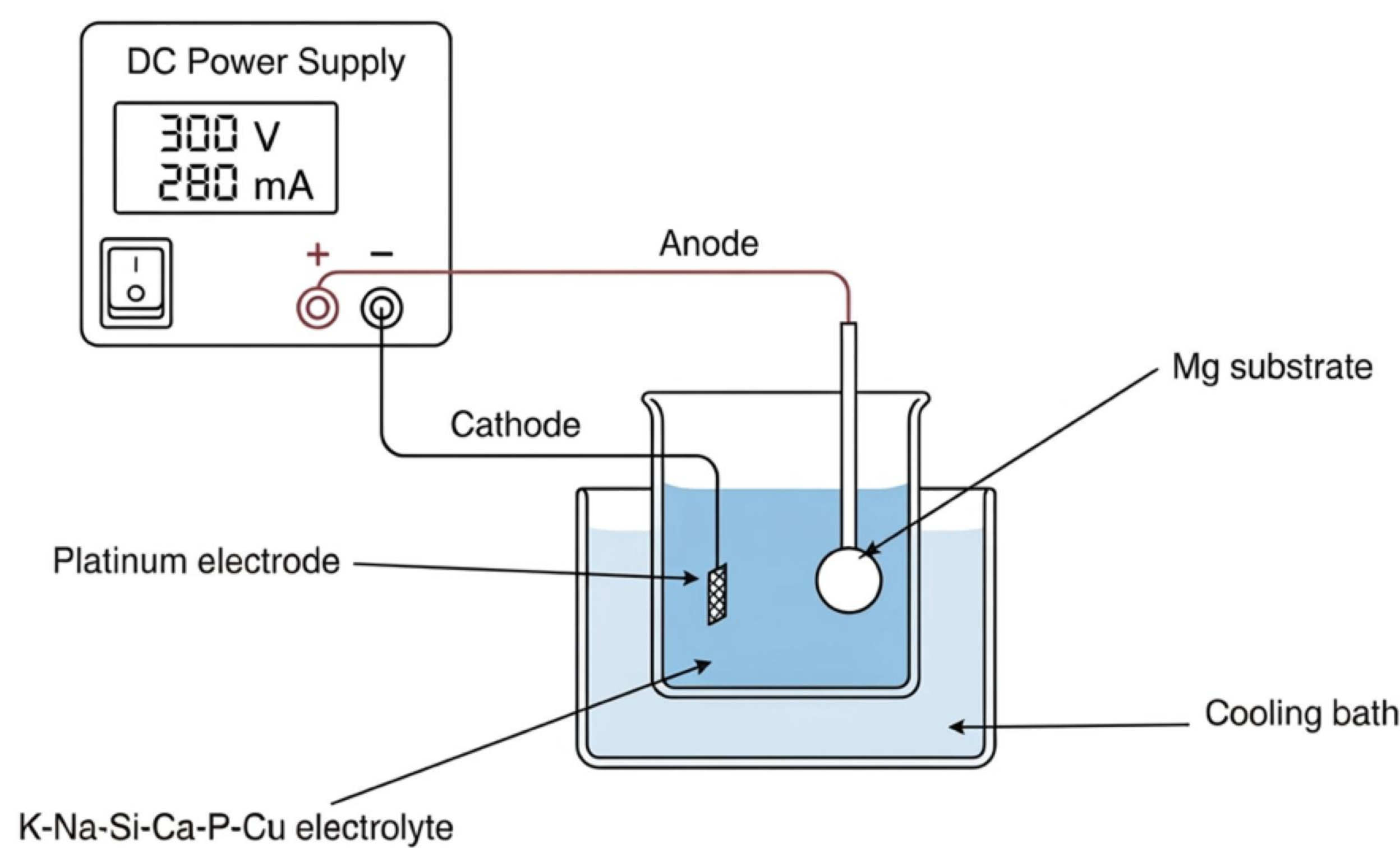


Figure 1. Experimental setup for MAO treatment of pure magnesium.

Characterization:

- **SEM** (Tescan Vega G4): morphology and pore structure;
- **EDS** (Oxford Instruments): elemental distribution and chemical composition;
- **XRD** (Malvern Panalytical Empyrean): phase composition;
- **3D Optical Profilometry** (S neox 090): surface topography (Sa, Sk, Spk, Sv, Is);
- **Coating Thickness** (FMP 10–20): coating thickness measurements;
- **Scratch Testing** (Step 500): coating adhesion and critical loads (LC₁, LC₂, LC₃);
- **Indentation** (NHT³ module, Step 500): hardness (H), elastic modulus (E), H/E, H³/E²;
- **Contact Angle & SFE** (Attension Theta Life): wettability and surface free energy (OWRK).

CONCLUSIONS

- MAO treatment successfully produced multifunctional oxide coatings on pure magnesium using a Ca/P/Si/Cu-containing electrolyte.
- Applied voltage was identified as the dominant parameter governing coating morphology, topography, thickness, mechanical properties, and wettability. Increasing voltage promoted coating growth, surface development, and improved mechanical performance.
- All MAO coatings exhibited enhanced surface characteristics compared with untreated magnesium, including higher hardness, increased surface free energy, and pronounced hydrophilicity.
- The results demonstrate that electrical parameters can be used to tailor MAO coatings toward different functional profiles, including enhanced mechanical durability at higher voltages and increased incorporation of bioactive elements under selected intermediate conditions.

FUTURE WORK

1. Corrosion testing under near-physiological conditions to evaluate coating stability and degradation behavior.
2. *In vitro* biological studies to assess cytocompatibility, cell adhesion, proliferation, and mineralization potential.
3. Antibacterial evaluation to determine the effectiveness of Cu-containing MAO coatings against bacterial adhesion and biofilm formation.

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REFERENCES

- [1] Makurat-Kasprolewicz B. et al. (2023). Recent advances in electrochemical surface treated titanium and its alloys for biomedical applications: A review of anodic and plasma electrolytic oxidation methods. *Materials Today Communications*, 34, 105425.
 - [2] Makurat-Kasprolewicz, B. et al. (2025). Frequency-Controlled AC-MAO Coatings with Ca, P, and Se on Magnesium: Toward Tailored Surfaces for Biodegradable Implants. *Materials*, 18, 5505.
 - [3] Dragan, W., et al. (2026). Structure-Property Control of MAO Coatings on Pure Magnesium in a Ca/P/Si/Cu-Containing Electrolyte via Voltage and Current. *MRS Communications* (accepted for publication).
- The results presented in this poster are based on the study reported in Reference [3], accepted for publication in *MRS Communications*.

RESULTS & DISCUSSION

Morphology and elemental incorporation

MAO treatment generated porous oxide coatings with voltage-dependent surface development. EDS confirmed the successful incorporation of Ca, P, Si, and Cu from the electrolyte. The highest Ca and P contents were observed for specimen 280_350, indicating favorable conditions for bioactive-element incorporation. Figure 2 shows EDS elemental maps demonstrating the distribution of incorporated elements within the coatings.

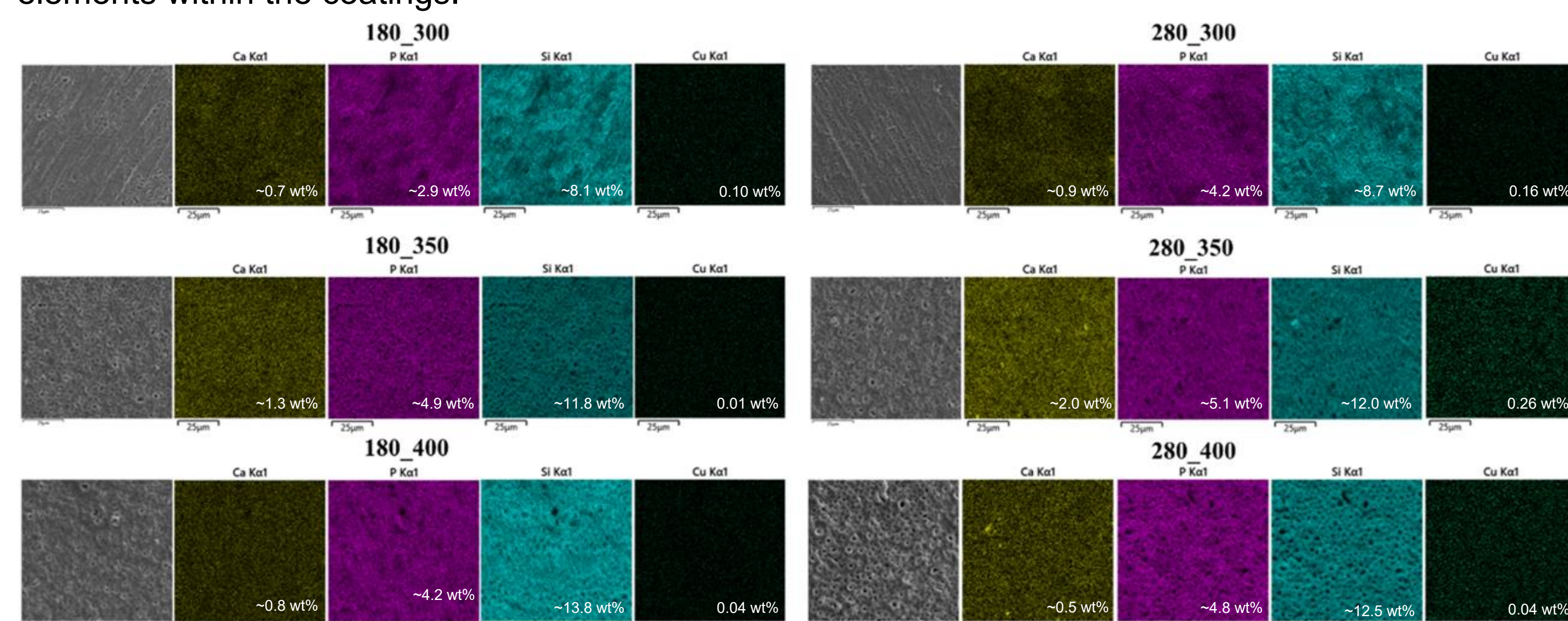


Figure 2. SEM images and EDS elemental maps of Ca, P, Si, and Cu for MAO coatings produced under different electrical parameters

Topography and coating thickness

Voltage was the dominant parameter controlling coating growth. Coating thickness and roughness increased with increasing voltage (Table 2), with the thickest coatings formed at 400 V. Enhanced micro-discharge activity at higher voltages resulted in more developed surface topographies (Figure 3).

Table 2. Topographical, thickness, and mechanical parameters of specimens.

Specimen	Sa [µm]	Is (%)	d (µm)	H (GPa)	E (GPa)
Mg	0.21±0.005	3.73±0.50	-	0.76±0.19	43.28±4.00
180_300	0.22±0.005	18.44±11.19	0.97±0.12	1.81±1.20	45.23±8.83 *
180_350	0.36±0.009 *	72.36±15.10 *	2.47±0.11	2.91±0.79 *	58.05±8.27 *
180_400	0.57±0.005 *	77.56±2.72 *	4.37±0.11 #	3.47±0.68 *	73.62±13.33 *
280_300	0.25±0.009 *,#	6.60±0.60	0.57±0.14	1.57±0.44	40.35±7.43 *
280_350	0.40±0.000 *,#	16.60±1.76 #	2.41±0.16	2.69±0.61 *	56.61±7.46 *
280_400	0.59±0.005 *,#	45.71±3.63 *,#	4.53±0.26	3.55±1.05 *	75.61±14.34 *

Sa – roughness; Is – isotropy; d – coating thickness; H – hardness; E – elastic modulus. Data are presented as mean ± SD. * p < 0.05 vs Mg; # p < 0.05 vs the corresponding 180 mA specimen.

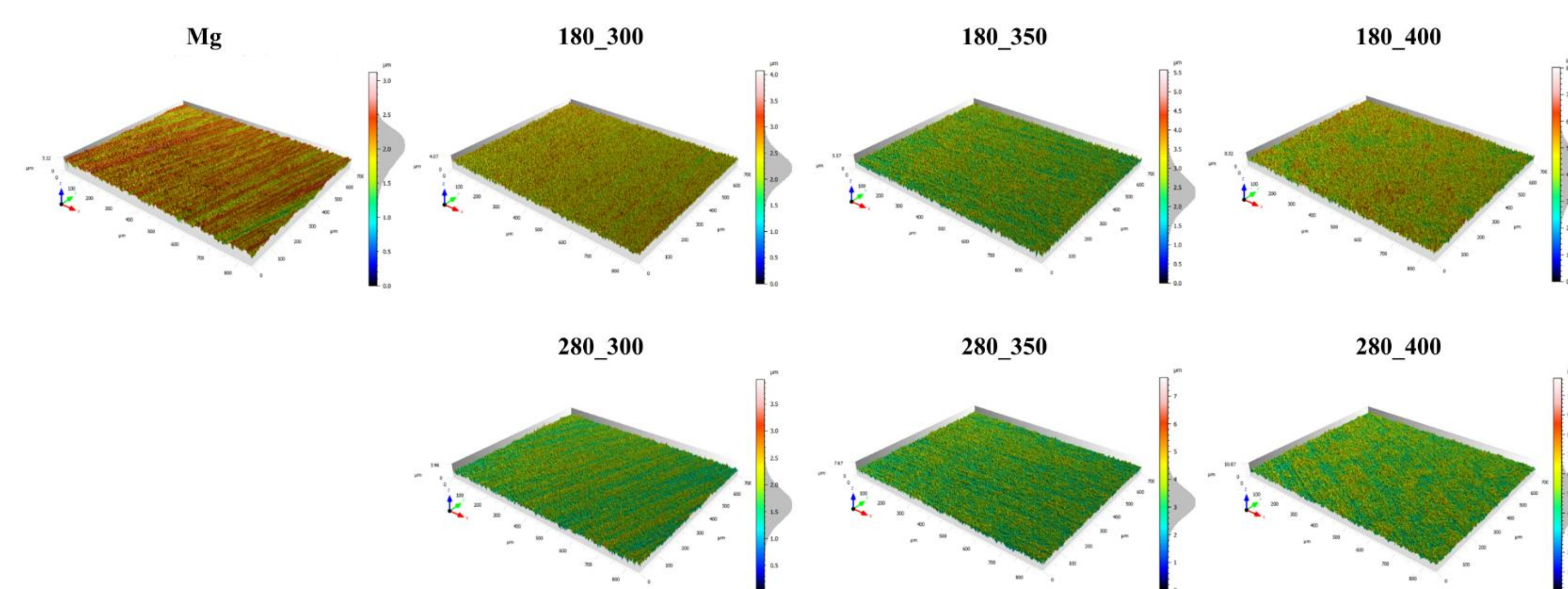


Figure 3. Three-dimensional surface topographies and corresponding peak-valley height distribution histograms of uncoated Mg and MAO-coated specimens.

Mechanical and adhesive properties

MAO coatings exhibited higher hardness than untreated magnesium (Table 2). Both hardness and elastic modulus generally increased with increasing voltage, indicating enhanced load-bearing capacity of the coatings. A similar trend was observed for scratch resistance (Figure 4), with the highest critical loads recorded for specimen 180_400. This coating exhibited the greatest resistance to mechanical damage, indicating superior mechanical durability and adhesion performance.

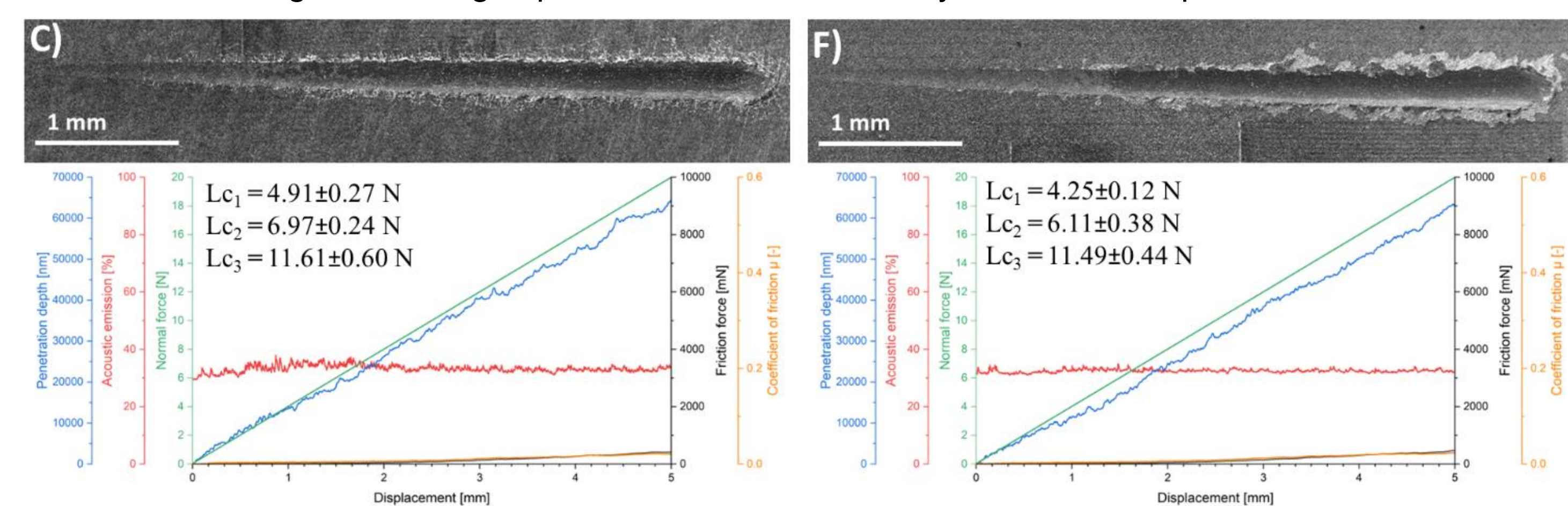


Figure 4. Representative scratch-test results for selected MAO-coated specimens: C) 180_400 and F) 280_400. SEM images of scratch tracks (40×) are shown together with the corresponding penetration depth, acoustic emission, friction force, and coefficient of friction as a function of normal load.

Wettability and surface free energy

All MAO coatings exhibited pronounced hydrophilicity and higher surface free energy compared with untreated magnesium (Table 3). Among the investigated coatings, specimen 280_300 showed the lowest water contact angle and the highest surface free energy, reflecting the strongest surface affinity toward polar liquids.

Table 3. Wettability and surface free energy (SFE) of the investigated specimens determined from contact angle (CA) measurements using a polar liquid (water, H₂O) and a non-polar liquid (diiodomethane, CH₂I₂).

Feature	Mg	180_300	180_350	180_400	280_300	280_350	280_400
CA for H ₂ O (°)	51.86±13.56	24.13±9.25 *	24.96±6.26 *	20.56±3.83 *	15.19±0.99 *	22.95±5.07 *	31.36±3.06 *
CA for CH ₂ I ₂ (°)	51.86±13.56	50.35±3.36	45.88±5.96	45.99±7.27	40.17±1.46	47.82±1.95	44.67±6.02
SFE (mJ/m ²)	56.66±8.02	68.69±27.82	69.53±3.22	71.48±1.48	74.39±0.24	63.08±13.47	65.98±2.21

* p < 0.05 vs Mg