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Application of EPR spectroscopy in the assessment of structural changes in powder paints under the influence of laser radiation

Piotr Pisiak, Bogumił Cieniek, Ireneusz Stefaniuk

Faculty of Exact and Technical Sciences
Institute of Materials Engineering
University of Rzeszow
e-mail: ppisiak@dokt.ur.edu.pl

Abstract

The aim of the study was to analyze the effect of laser irradiation on the defect structure in powder coatings using electron paramagnetic resonance (EPR) spectroscopy. Two types of powder paints were examined, both polyester-based: A – red, and B – black. Measurements were conducted before and after irradiation with a laser (532 nm, 300 mW), in the temperature range of 300–500 K. Significant changes in the EPR signal were observed in the sample after laser irradiation, suggesting activation and/or migration of paramagnetic centers.

Introduction

Electron paramagnetic resonance spectroscopy is a sensitive technique used to detect unpaired electrons, structural defects, radicals, or transition metal ions [1]. The method enables the determination of the electronic structure and defect dynamics in a magnetic field [2]. EPR is non-invasive, allows studies over a wide temperature range and enables the assessment of material stability under external influences. The intensity and shape of the EPR signal are strongly dependent on temperature and instrument settings, including microwave attenuation [3]. In this study, a Bruker Elexsys E580 spectrometer equipped with an ER 4131VT variable temperature unit was used, which allowed for signal acquisition under stable and controlled temperature conditions [3]. The ER 4131VT unit made it possible to carry out measurements across a broad temperature range while maintaining stable experimental conditions.

Measurement Metodology

The following equipment and parameters were used in the experiments:

Laser: green semiconductor laser, wavelength 532 nm, power 300 mW (class 3B)
Spectrometer: Bruker Elexsys E580 with ER 4131VT variable temperature unit
Microwave attenuation: 10 dB;
Modulation amplitude: 5 dB.

Each sample was measured before and after exposure to laser. Changes in the EPR signal were analyzed as a function of temperature. In addition, the measurement methodology:
– before irradiation (0 s),
– after 150 seconds of exposure,
– after 150 s followed by 5 minutes of relaxation,
– after 150 s followed by 10 minutes of relaxation.

Results

Sample A: The EPR spectra recorded before laser exposure showed a single signal located within the magnetic field range of 3200–3350 G. This signal maintained a consistent shape and intensity across the entire temperature range, indicating the presence of stabilized paramagnetic centers, likely associated with lattice defects and oxide impurities [1]. After exposure to laser radiation a noticeable increase in EPR signal intensity was observed, especially at temperatures above 400 K. Additionally, the signal broadened and slightly split, which may indicate the emergence of new paramagnetic centers or transient structural changes in the local defect environment [3]. These effects were particularly visible in the 420–450 K range, where differences compared to pre-irradiation measurements became most pronounced. This may suggest the activation of Frenkel-type defects (related to the displacement of atoms into interstitial positions) or the rearrangement of electrons trapped in metastable states. No complete disappearance of the original signals was observed, which suggests the coexistence of both original and induced structures [8].

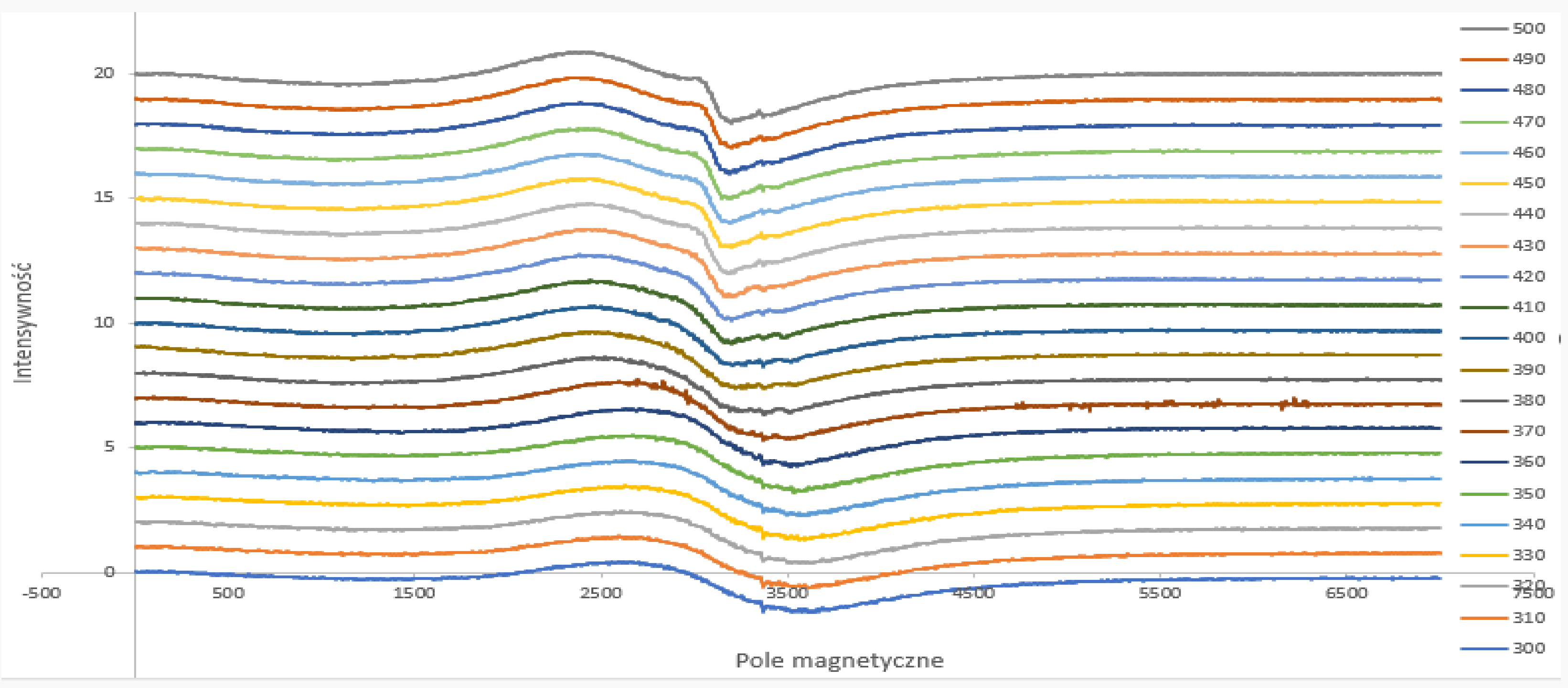

Figure 1. Sample A – red polyester-based powder coating (before irradiation)

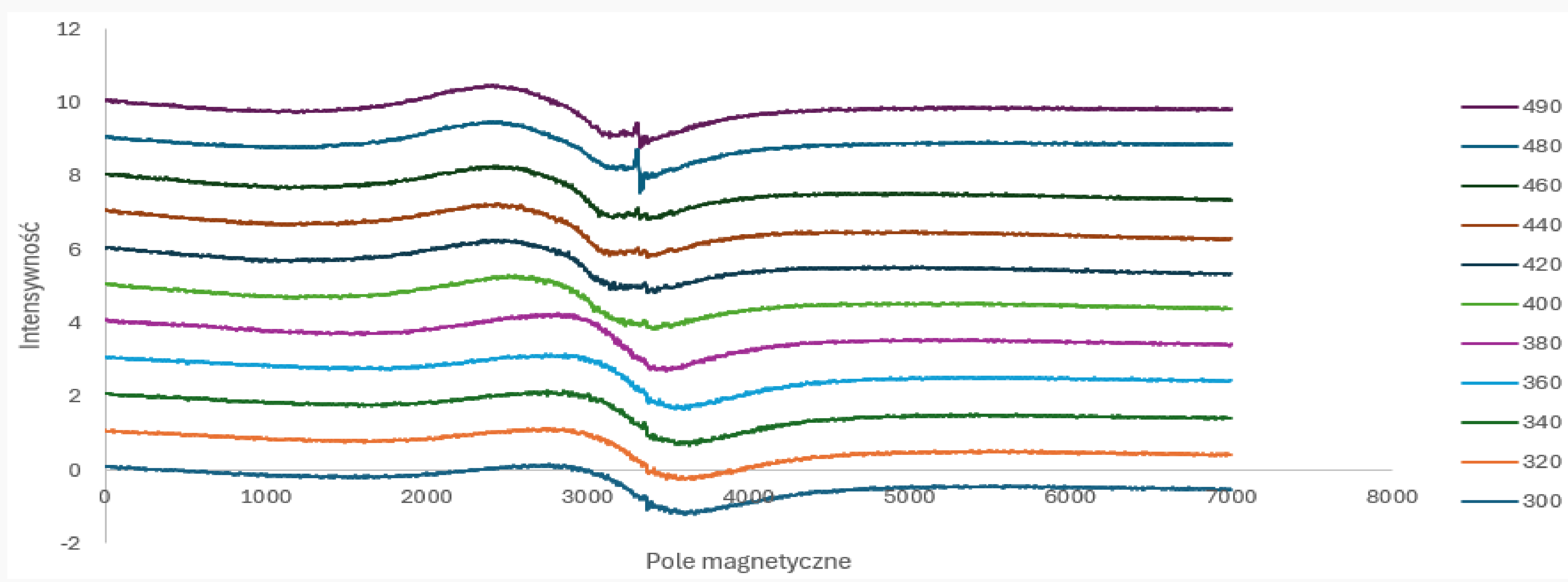

Figure 2. Sample A – red polyester-based powder coating (after irradiation)

Sample B: At 300 K, a single signal centered around 1900 Gs was observed. The pre-irradiation measurement showed moderate signal intensity, indicating the presence of permanent defects in the paint structure [4]. After 150 seconds of laser exposure, a significant increase in signal intensity was recorded, which may indicate photoexcitation and the formation of new trap states. Additional measurements taken 5 and 10 minutes after the exposure showed a systematic decrease in intensity, suggesting partial relaxation of the electronic structures. These changes indicate the presence of metastable states that gradually decay over time [1].

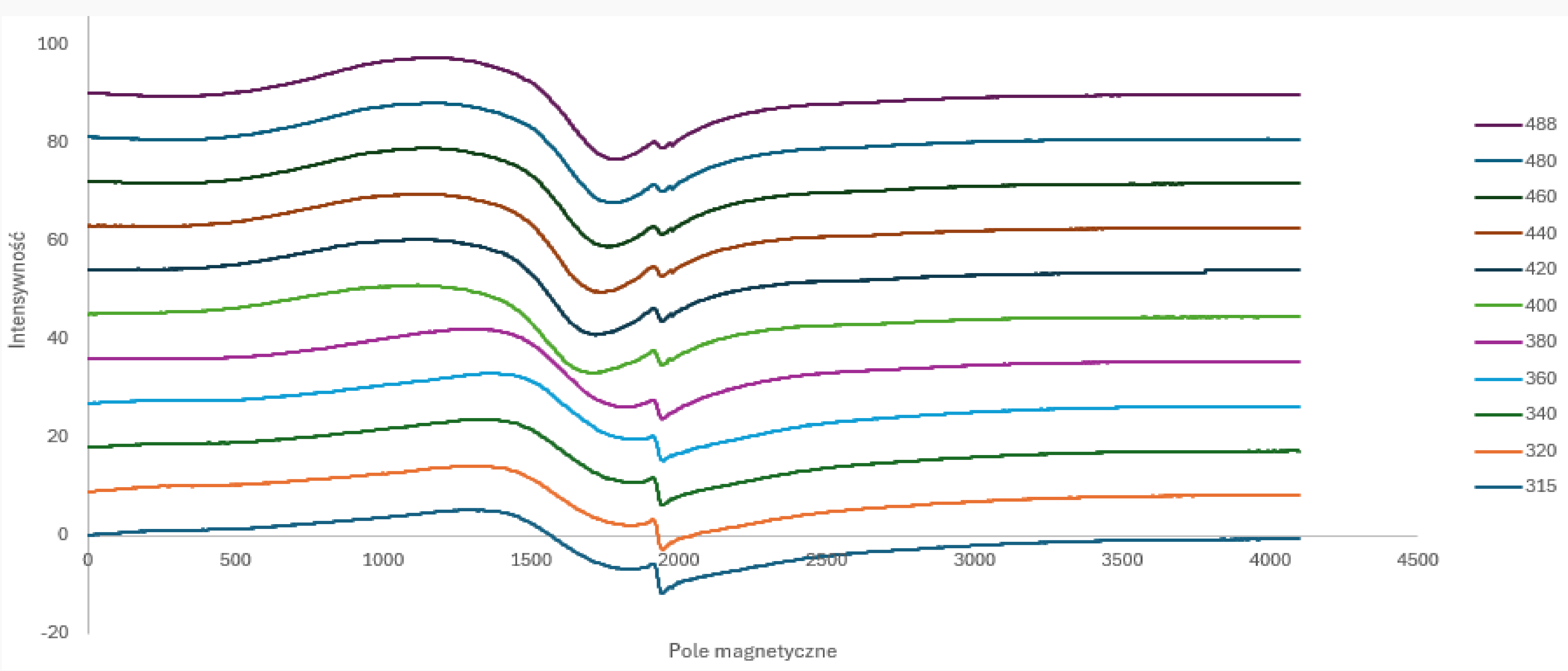

Figure 3. Sample B – black polyester-based powder coating (after irradiation

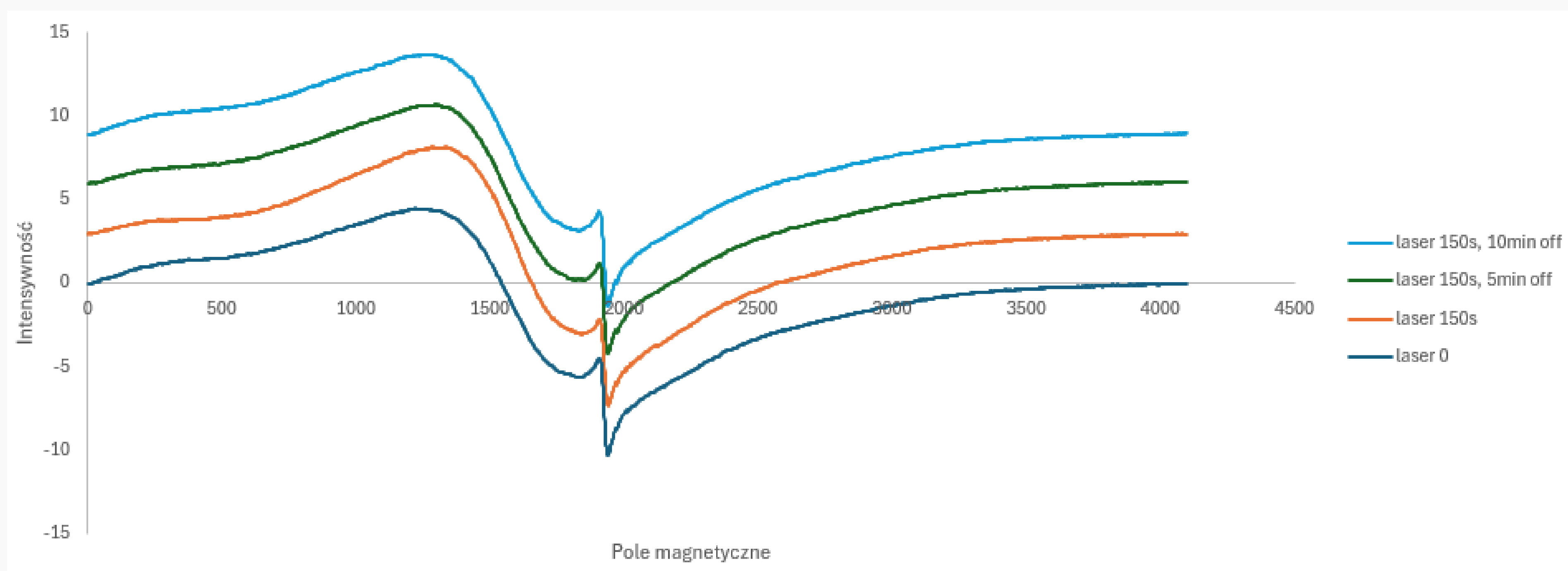

Figure 4. Sample B – black polyester-based powder coating (after irradiation)

Conclusions

- We confirms the high sensitivity of EPR spectroscopy in detecting subtle structural changes in powder coatings induced by laser radiation.
- In the case of sample A, a particularly strong EPR response was observed in the 420–450 K range after laser exposure, indicating increased mobility or reorganization of paramagnetic centers.
- For sample B, measurements showed that some changes undergo relaxation, suggesting the involvement of metastable states.
- The EPR method can be successfully used to evaluate the chemical and structural stability of materials exposed to increased environmental stress, including radiation, high temperatures, and pressure fluctuations.

Industrial Significance and Practical Applications

Due to its sensitivity to changes in the local environment of unpaired electrons, EPR spectroscopy can serve as an effective tool for monitoring the durability of coating materials and verifying their resistance to radiation, temperature, and other aging-accelerating factors. It can also be applied in quality control of industrial materials during production and after long-term use. EPR can help identify early signs of degradation, allowing for preventive maintenance and improved product reliability.

References

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