Paper published in
International Journal of Adhesion and Adhesives, Volume 139, 2025, 103981,
https://doi.org/10.1016/j.ijadhadh.2025.103981.
(https://www.sciencedirect.com/science/article/pii/S014374962500048X)

Impact of Thermal and Humidity Conditions on Structural Epoxy Adhesives During Medium-Term Exposure

Marcin Kujawa[1], Piotr Paczos[2], Łukasz Smakosz[1], Adam Piasecki[2], Faizullah Jan[1], Karol Winkelmann[1], Viloletta Konopińska-Zmysłowska[1], Victor A. Eremeyev[3]

[1] *Gdańsk University of Technology*
G. Narutowicza 11/12, 80-233 Gdańsk Poland
[2] *Poznań University of Technology*
Jana Pawla II 24, 61-139 Poznań Poland
[3] *University of Cagliari*
Via Marengo 2, 09123 Cagliari Italy

Abstract

An experimental program was undertaken to evaluate the performance characteristics of two commercially available two-component structural epoxy resin adhesives under varying thermal and humidity conditions, including elevated temperatures, hygrothermal conditioning, and freeze-thaw cycles. The adhesives investigated, 3M Scotch DP490 and DP125 Gray, were selected for their relevance in practical applications. A medium-term exposure period was adopted to more accurately simulate real-world service conditions. The characterization of these adhesives was conducted using scanning electron microscopy, standard tensile tests, and Shore hardness tests. DP490, a high-stiffness epoxy, is primarily used for bonding small, unpainted metal components, as well as metal to glass, ceramics, and stone. DP125 Gray is a more flexible system, typically employed for bonding metal to glass, glass to plastics, and painted or powder-coated metals. This study provides an analysis of the strengths and limitations of DP490 and DP125 Gray adhesives, with a focus on their performance under various conditioning scenarios relevant to civil and mechanical engineering applications.

Keywords: epoxy adhesive properties, elevated temperature conditioning, hygrothermal conditioning, freeze-thaw conditioning

1. Introduction

The primary focus of research on structural adhesives is mainly on epoxy resins [1]. This is due to the unique properties of epoxy adhesives, known for their exceptional bonding capabilities with variety of materials. Epoxy adhesives can be categorized based on various criteria. The fundamental classification of epoxy adhesives is based on their chemical composition, leading to their division into two categories: pure epoxies (made primarily of epoxy resins, these adhesives offer strong bonding and high temperature resistance) and more popular modified epoxies (these contain additives or modifiers to enhance certain properties like flexibility, toughness, or cure time) [2, 3]. Another difference is due to the different mechanism of curing the adhesive. There are two types of epoxy with respect to the curing mechanism: one-component epoxy resin (pre-mixed and require heat to initiate the curing process) and two-component epoxy resin (components are mixed immediately before use, cure at room temperature, and provide flexibility in application timing). Epoxy adhesives can also be classified with regard to their viscosity into: liquid epoxy (low viscosity, suitable for penetrating small gaps and cracks) and thixotropic or paste epoxy (higher viscosity, good for applications requiring gap filling or vertical application without dripping). One also classifies epoxies based on their curing speed into two types: fast-curing (designed for quick assembly, these cure in minutes or hours) and slow-curing epoxy (have longer working times, suitable for complex or large-scale applications). Epoxy adhesives are also differentiated by their temperature resistance into standard (perform well under normal temperature conditions) and high-temperature (formulated to withstand extreme heat, maintaining bond integrity at elevated temperatures) adhesives.

At this point, it is also important to mention the consideration of environmental care within the framework of the Stockholm Convention on Persistent Organic Pollutants (POPs). The convention addresses PFASs (Per- and Polyfluoroalkyl Substances), which are related to the reduction in the production of materials using fluorine—an element not naturally occurring in nature. Fluorine, as an element, is rarely an ingredient directly added to epoxies. However, some substances used to modify the properties of epoxies, such as hardeners or accelerators, may contain fluorine atoms in their chemical structure. For example, modifiers containing fluorine may be used in specialized applications where exceptional chemical or thermal resistance is required. However, this is not standard practice for all epoxy adhesives, but rather an exception used under specific conditions.

The systematic use of structural adhesives in industry began in the mid-20th century and continues today, with epoxy adhesives being utilized in various engineering fields to bond and reinforce structures made of wood, steel, brick, and concrete [4, 5]. The long-term performance and durability of bonded structures are critical to ensuring structural safety, as these structures are exposed to environmental conditions that can limit their functionality due to material degradation. Throughout the lifespan of a structure, interactions may occur between various physical and environmental factors, such as when a material or structural element is simultaneously exposed to sustained loads, changing temperatures, and moisture. This underscores the importance of accurately determining the actual performance of structural adhesives subjected to different environmental conditions.

One of the main environmental factors limiting the application range of structural epoxy adhesives is their temperature sensitivity. The mechanical properties of epoxy adhesives, like strength and stiffness, are significantly affected by the conditions under which they cure, involving the cross-linking of polymer chains [6, 7]. Therefore it is advisable for certain types of epoxy adhesives to undergo a final curing stage at higher temperatures than the initial cure, which could potentially improve mechanical properties even with minimal increases in curing. When an adhesive reaches a certain temperature equal to or higher than its glass transition temperature [8], there is a sudden change in its properties: it transitions from a hard and relatively brittle state to a rubber-like state. Therefore, the value of the glass transition temperature limits the use of the adhesive with respect to the maximum operating temperature. The phenomenon of post-curing is also observed when the epoxy's temperature momentarily exceeds the glass transition temperature, subsequently increasing the glass transition as well. Additionally, when the adhesive is cooled from temperatures above the glass transition to below it, its mechanical properties are completely restored.

Other key environmental elements that can cause changes in properties through physical and chemical changes are humidity and water penetration [9, 10, 11, 12]. The problem is particularly significant for epoxy adhesives because they absorb water due to the presence of polar groups that attract water molecules. Water absorption by epoxy adhesives leads to several adverse effects: water absorbed by the adhesive can cause it to swell, leading to weakened adhesion bonds and reduced shear and tensile strength, and at negative temperatures, it can cause the adhesive to burst; water can induce hydrolysis processes in which the chemical bonds in the epoxy matrix are broken, causing further deterioration of the adhesive's mechanical and chemical properties; water absorption can affect the glass transition temperature of the epoxy adhesive, altering its properties at different operating temperatures; and water can also contribute to the corrosion of metal substrates to which the epoxy adhesive has been applied, further weakening the adhesive bond. Thus, water can irreversibly change the mechanical behavior of epoxy resins. As Lettieri et al. note in their paper [11], the effects of water absorption are detrimental only at humidity levels higher than 75%, which is unfortunately not uncommon in real-world situations. For example, in Central and Eastern Europe, these levels range between 68-85% depending on the season. Particular care should be taken with epoxy resin because of its high susceptibility to moisture during its setting, that is, before curing. This is because it is particularly hygroscopic at this stage (Hygroscopic polymers also include acrylonitrile-butadiene-styrene (ABS), acrylic, polyethylene terephthalate (PET), polybutylene terephthalate (PBT), polyurethane (PU), polycarbonate (PC), and others, such as nylon, while non-hygroscopic polymers include polyvinyl chloride (PVC), polypropylene (PP), polystyrene (PS), polyethylene (PE), and others.). Due to the sensitivity of epoxy adhesives to water, protecting adhesives from moisture and humidity is an important aspect [13]. Of course, in the case of adhesively bonded adherends, it is relevant only at the edges of the joints between bonded elements, but it is worth remembering.

The next important impact is the effect of hygrothermal aging on the glass transition temperature, which has been investigated by various researchers [9, 12]. For epoxy resin, it is understood that the glass transition temperature is connected to the crosslink density. When the crosslink density decreases, the glass transition occurs at a lower temperature, and thus hygrothermal aging adversely affects the epoxy system.

In conclusion, the results of experiments conducted by many researchers have shown that the behavior and condition of adhesives can be significantly influenced by environmental factors.

For a summary and details of research and an overview of epoxy adhesives, see Tabs 1 and 2. Table 1 summarizes the mechanical properties of epoxy adhesives mostly at room temperature (23°C) and no conditioning, such as: tensile modulus of elasticity E, tensile failure strength σ_f, strain ε_f corresponding to tensile failure strength, shear failure strength τ_f, strain corresponding to shear failure strength γ_f, Poisson's ratio ν, shear modulus G. Table 2 summarizes the mechanical properties of epoxy adhesives – tensile modulus of

elasticity E, tensile failure strength σ_f, and glass transition temperature T_g – subjected to various environmental exposures. In addition, Fig. 1 summarizes selected epoxy adhesives, indicating how tensile strength values and corresponding Young's modulus values change for previously unconditioned adhesives. Based on the available experimental results, an increase in strength and Young's modulus values is usually observed for series subjected to elevated temperature cycles. In contrast, a decrease in strength and Young's modulus values was observed in samples subjected to aging cycles involving immersion in water (hydroaging) and exposure to water vapour (hygroaging). For epoxy adhesives, Young's moduli at room temperature (23°C) range from 0.082 GPa (3M Scotch SBT9244) [14] to 13.289 GPa (Sika Sikadur 30 LP) [15] (Fig. 1). The values of Young's moduli generally decrease with increasing temperatures during the tests [16, 17, 18, 19, 20]. However, the values may increase when the tests are performed at sub-zero temperatures [16, 17]. Young's modulus values are closely correlated with tensile strength values (Tab.1). When one increases due to varying aging conditions, the other also increases, and vice versa. The highest tensile strength value for unconditioned adhesives analyzed is 93.40 MPa (Mapei Epojet) [11], and the lowest is 12.63 MPa (Huntsman Araldite 2015) [21, 22] (Fig. 1). The highest value for shear stress was 61.00 MPa (Huntsman Araldite MY750) [23], and the lowest was 17.90 MPa (Huntsman Araldite 2015) [21, 22].
It is important to recognize that each adhesive comes with its own set of pros and cons, and selecting the appropriate adhesive is contingent upon the specific requirements of the application. Adhesives exhibiting high strength are best suited for applications demanding substantial mechanical strength, whereas those with a high Young's modulus are more suitable for scenarios requiring enhanced structural stiffness.

As demonstrated by the literature review (Tab. 2), available information regarding the most commonly used epoxy adhesives in civil and mechanical engineering is still relatively limited. This is particularly true for studies where adhesives were preconditioned prior to testing [7, 24]. Thus, the primary aim of this study is to assess commercial structural epoxy adhesives under thermal, humidity, and moisture conditions. To achieve this, a specific experimental program was conducted, wherein epoxy samples underwent exposure to various thermal-humidity conditions (including high temperatures, thermal-humidity cycles, and freeze-thaw cycles) for periods of 7, 14, and 28 days. Typically, aging tests are conducted over long periods (approx. 2500 hours, which is 104 days) or short periods (approx. 96 hours, which is

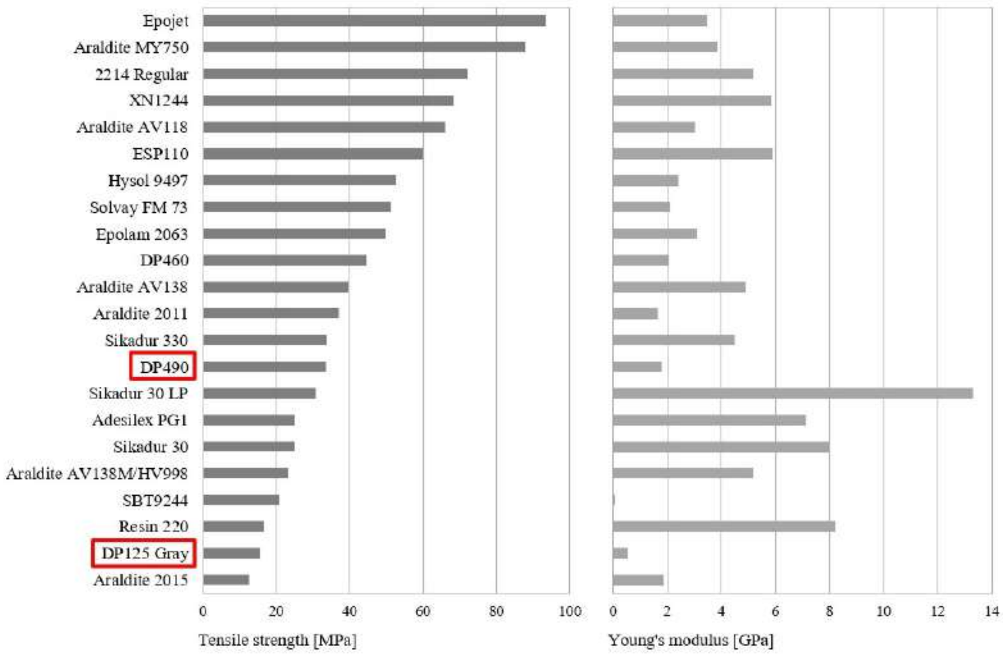


Figure 1: Tensile strength and Young's modulus for selected epoxy adhesives without preconditioning

4 days). The medium-term exposure durations of 7, 14, and 28 days were chosen to more closely resemble real working conditions. However, the climate stress conditions assumed in the study are still quite extreme, especially for Europe. Characterization was performed using tree methods: scanning electron microscopy (SEM), standard tensile tests, and Shore hardness tests. For research using SEM, we will focus on examining and analyzing the surface and near-surface areas of the adhesives tested, as well as the chemical composition of the adhesives. In standard tensile tests, the breaking strength was determined for both conditioned and unconditioned specimens, while the tensile modulus and Poisson's ratio were determined only for unconditioned specimens. In the analysis of Shore hardness, attention was focused on the relationship between changes in hardness and parameters related to strength.

Table 1: Mechanical properties of selected epoxy adhesives without preconditioning

Ref.	Name	E [GPa]	σ_f [MPa]	ε_f [%]	τ_f [MPa]	γ_f [%]	ν [-]	G [GPa]
2003 [25]	Infinity Bond ESP110	5.900	60.00	2.0	–	–	0.40	2.110
	3M Scotch DP490	1.800	33.45	5.3	–	–	0.40	0.640
2006 [26]	3M Scotch 2214 Regular	5.170	72.00	2.0	–	–	0.35	1.910
2007 [16]	Master Bond	3.450	–	–	38.60	20.7	0.36	1.270
	Supreme 10HT	4.890/-55C			60.57	16.4	0.36	1.800
		2.180/100C			18.19	45.0	0.36	0.800
		0.040/200C			2.77	28.0	0.50	0.010
2009 [17]	Huntsman	3.010	65.90	5.9	25.55	29.2	0.38	1.090
	Araldite AV118	3.620/-40C	69.70	2.5				
		2.450/60C	44.80	10.5				
		1.850/80C	29.70	12.0				
2011 [23]	Huntsman Araldite MY750	3.850	88.00	3.2	61.00	15.0	0.35	1.330
2011 [21] 2018 [22]	Huntsman Araldite AV138	4.890	39.45	1.2	30.20	7.8	0.35	1.560
	Huntsman Araldite 2015	1.850	12.63	4.8	17.90	43.9	0.33	0.560
2012 [18]	Nagase	5.870	68.23	1.5	31.61	8.1	0.35	2.150
2014 [19]	XN1244	4.170/100C	45.16	1.9				
		0.070/150C	6.49	13.7				
		0.040/200C	1.44	3.3				

Table 1: Mechanical properties of selected epoxy adhesives without preconditioning

Ref.	Name	E [GPa]	σ_f [MPa]	ε_f [%]	τ_f [MPa]	γ_f [%]	ν [-]	G [GPa]
2013 [14] 2015 [27]	3M Scotch SBT9244	0.082	20.90	94.5	–	–	0.35	0.030
	3M Scotch DP460	2.080	44.60	4.3	–	–	0.38	0.750
2014 [28]	Henkel Terokal 5045	0.437	–	–	20.00	11.3	0.38	0.160
	Henkel Hysol EA9313	2.274	–	–	27.60	8.0	0.36	0.840
2015 [29]	Henkel Hysol 9497	2.400	52.60	–	–	–	0.30	0.920
2015 [27]	Solvay FM 73	2.09	51.05	11.0	–	–	0.35	0.770
2018 [30]	Sika Sikadur 330	4.500	33.80	0.8	28.30	1.9	0.25	1.800
2019 [20]	S&P	8.200	16.60	0.1	25.10	0.1	–	4.610
	Resin 220	6.320/35C	12.20	0.1	21.80	0.1		2.950
		5.970/42.5C	12.80	0.1	18.40	0.1		2.560
		0.180/50C	5.20	0.8	8.10	41.1		0.050
		0.083/70C	2.50	0.4	4.10	28.3		0.040
		0.073/90C	1.80	0.3	2.30	0.7		0.004
		0.039/120C	1.60	0.2	2.00	0.3		0.003
2019 [31]	Mapei Adesilex PG1	7.110	24.89	0.4	13.70	–	0.26	2.720
2022 [32]	3M Scotch 7260B/A	3.000	–	–	33.50	3.0	–	–
	3M Scotch 7240B/A	1.500	–	–	29.40	–	–	–

Table 2: Mechanical properties of selected epoxy adhesives with preconditioning

Ref.	Name & Effects	Group of samples	E [GPa]	σ_f [MPa]	T_g [°C]
2005 [9]	DGEBA/DDA	Reference/Dry	3.174	22.98	–
	Effects of hygrothermal aging	Sorption	2.239	–	–
	Deionized water 85°C/85%RH	Desorption	2.891	–	–
	~50 months	Resorption	1.821	–	–
	Effects of moisture aging	Sorption	–	16.29	–
	Deionized water 23°C	Desorption	–	20.57	–
	~50 months	Resorption	–	10.56	–
2008 [10]	Sika Sikadur 30	Reference/Dry	7.970	24.80	103.0
	Effects of moisture aging	Deionized water 23.0°C	4.450	14.50	78.1-96.9
	~24 months	Deionized water 37.8°C	4.060	13.85	84.7-111.9
		Deionized water 60.0°C	2.220	7.76	85.5-118.4
		Salt solution 23.0°C	4.780	15.94	79.1-101.9
		Alkali solution 23.0°C	3.540	12.00	–
2012 [11]	Mapei Epojet	Reference/15%RH	3.450	93.40	57.1
	Effects of humidity	55%RH	3.160	97.90	51.9
	Deionized water 23.0°C	75%RH	3.270	105.70	51.5
	~1 month	100%RH	3.000	93.90	49.4
	Effects of moisture	Deionized water 23.0°C	2.940	91.40	51.7
	~1 month				
2014 [6]	Huntsman Araldite 2011	Reference 23.0°C/55%RH	1.670	37.10	64.8
	Effects of cure temperature	40.0°C	1.779	44.90	67.4
	~3.5 months	60.0°C	1.712	40.40	73.4
		80.0°C	1.664	36.60	70.0
		100.0°C	1.609	34.40	62.3
		120.0°C	1.520	29.90	56.8
	Huntsman Araldite AV138M/HV998	Reference 23.0°C/55%RH	5.183	23.10	75.1
	Effects of cure temperature	40.0°C	5.312	35.10	77.9
	~3.5 months	60.0°C	5.459	42.70	82.8
		80.0°C	5.566	50.70	98.6
		100.0°C	5.612	52.80	102.9
		120.0°C	5.541	44.20	97.2

Table 2: Mechanical properties of selected epoxy adhesives with preconditioning

Ref.	Name & Effects	Group of samples	E [GPa]	σ_f [MPa]	T_g [°C]
2014 [6]	Sika Sikadur 30 LP	Reference 23.0°C/55%RH	13.289	30.80	69.7
	Effects of cure temperature	40.0°C	13.578	33.30	86.9
	~3.5 months	60.0°C	14.045	36.60	104.0
		80.0°C	14.269	40.90	113.5
		100.0°C	14.547	43.50	120.6
		120.0°C	14.638	44.90	125.5
		140.0°C	14.318	40.90	120.1
2014 [12]	Axson Technology Epolam 2063	Reference	3.100	50.00	168.6
	Effects of hygrothermal aging	~3 months	–	–	153.7
	Pre-curing 12H 70°C/90%RH	~8 months	–	–	147.4
	Deionized water 70°C/90%RH/-40°C				
2016 [7]	S&P Resin 220				
	~0 months	Reference	7.150	22.00	–
	~8 months	Reference	–	–	47.2-59.6
	~16 months	Reference	6.660	20.81	53.6-60.3
	Effects of moisture aging				
	~8 months	Deionized water	4.100	13.63	49.1-59.6
	~16 months	Deionized water	3.540	12.94	46.2-60.5
	~8 months	Chlorides solution (3.5% NaCl)	4.730	15.33	42.4-57.1
	~16 months	Chlorides solution (3.5% NaCl)	4.390	15.05	45.2-60.9
	Effects of wet/dry aging				
	~8 months	Chlorides solution (3.5% NaCl)	5.450	16.59	50.2-59.1
	~16 months	Chlorides solution (3.5% NaCl)	5.220	16.56	50.4-62.0
	Effects of thermal aging				
	~4 months	-15°C/60°C	7.510	25.91	43.8-59.3
	~8 months	-15°C/60°C	7.640	27.32	48.6-63.1
	Effects of freeze-thaw aging				
	~4 months	-18°C/20°C	5.950	18.54	–
	~8 months	-18°C/20°C	5.560	17.28	41.5-56.7

2. Experimental tests

2.1. General characteristics of adhesives

The first tested adhesive is a two-component, structural epoxy thermosetting resin (3M Scotch DP490) of high stiffness. According to the manufacturer, the product provides a strong, durable bond with excellent resistance to heat and ambient conditions, withstanding temperatures up to 120°C. The adhesive is used to join small, unpainted metal parts (metal to metal), as well as unpainted metal to glass, ceramics, and stone and should be applied at temperatures of 40°C or below. The approximate shelf life of the adhesive after mixing at room temperature is 90 minutes, and the approximate time to achieve handling strength is 240-360 minutes. Full cure time is 7 days (168 hours). The adhesive has primarily found its applications in transportation, including the railroad, automotive, and aerospace industries. The shear strength of DP490 adhesive, according to the manufacturer's specifications, at different temperatures is: 24.00 MPa at -55°C, 30.00 MPa at 23°C, and 12.00 MPa at 82°C. The tensile strength is approximately 36.00 MPa (at 23°C). Generally, the adhesive is a black (Fig. 4a), thixotropic paste with a mild odor. Its density ranges from 0.97 to 1.1 g/cm^3 (at 23°C). The adhesive has a flash point of $\geq$ 93.3°C (test method: closed crucible). The adhesive's composition includes 2,2-bis[4-(2,3-epoxypropoxy)phenyl]propane (50-60%), MBS copolymer of methyl methacrylate/butadiene/styrene (10-20%), 1,4-bis(2,3-epoxypropoxy)methylcyclohexane (5-15%), glass fiber (1-5%), 3-(trimethoxysilyl)propyl glycidyl ether (0-1.5%), and titanium dioxide (0-1.5%). Contact with the adhesive can cause severe skin burns and eye damage, trigger allergic skin reactions, and induce drowsiness or dizziness. Notably, it can be highly toxic to aquatic life, potentially causing long-term adverse effects in water. Hazardous decomposition products such as aldehydes, carbon monoxide, carbon dioxide, and hydrogen chloride may be released during combustion. This material does not contain any substances that would be assessed as endocrine disruptors for human health. Unfortunately, the adhesive is not resistant to oxidants and acids; contact with oxidizing agents such as chlorine and chromic acid should be avoided.

The second tested adhesive is a two-component, structural epoxy resin (3M Scotch DP125 Gray) with a flexible structure. The adhesive is used to bond metal (aluminum alloys, steel, stainless steel, galvanized steel) to glass, glass to glass, and glass to plastics (ABS (Acrylonitrile-Butadiene-Styrene), acrylics (PMMA - Polymethyl Methacrylate), polycarbonates (PC), and nylon (PA - Polyamide)). It is also effective for bonding painted or powder-coated elements (metals, composite panels) to glass. The approximate shelf life of the adhesive after mixing at room temperature is 25 minutes, and the approximate time to achieve handling strength is 150 minutes. Full cure time is 7 days (168 hours). The adhesive is mainly used for general bonding applications across various industries. The shear strength of DP125 Gray adhesive, according to the manufacturer's specifications, at different temperatures is: 23.40 MPa at -55°C, 29.60 MPa at 23°C, and 2.80 MPa at 82°C. The tensile strength is approximately 22.75 MPa (at 23°C). Generally, the adhesive is a dark gray (Fig. 4b), controlled flow paste with a mild odor. Its density is approximately 1.05 g/cm^3 (at 23°C). The adhesive has a flash point of $\geq$94.0°C (test method: closed crucible). The adhesive's composition includes 2,2-bis[4-(2,3-epoxypropoxy)phenyl]propane (50-60%), polymer of epichlorohydrin with 4,4'-(1-methylethylidene)-biscyclohexanol (15-40%), kaolin (10-30%), dimethylsiloxanes and silicones, reaction products with silica (1-5%), and titanium dioxide (<0.5%). Contact with the adhesive can cause severe skin burns and eye damage, and trigger allergic skin reactions. This material does not contain any substances that would be assessed as endocrine disruptors affecting the environment; however, it is toxic to aquatic organisms and may cause long-term effects.

2.2. Specimens and environmental exposure conditions

An experimental study was performed to evaluate the properties of a structural epoxy adhesive under a range of aging actions, such as high temperatures, thermal-humidity cycling, and freeze-thaw cycles. In the first part of the study, for elevated temperatures, the samples were conditioned at 40°C, 60°C, and 80°C for durations of 7, 14, and 28 days. A total of nine samples were conditioned three times, with three samples at each temperature. The second part of the study, includes conditioning periods of 7, 14, and 28 days, involving a total of nine samples. The testing consists of four phases conducted within 24 hours: phase 1 lasts 4 hours at 40°C and 90% humidity, phase 2 lasts 8 hours at -20°C, phase 3 lasts 8 hours at 70°C and 90% humidity, and phase 4 lasts 4 hours at 20°C and 60% humidity. In the third part of the study, temperatures ranged from -20°C to +20°C/45%RH, with plateaus lasting 16 and 8 hours, respectively. Once more, a total of nine samples were conditioned, with three samples each for 7, 14, and 28 days. The study utilized an ESPEC Type LHU-114 dynamic climate chamber. For each conditioning scenario, the samples were tested no earlier than

28 days after exposure. During this time, the samples were stored at a room temperature of 23°C and approximately 55% relative humidity (RH). After exposure to various environmental conditions, the samples were examined using a scanning electron microscope, a strength testing machine, and a Shore durometer. The results were compared with the values obtained for reference samples.

2.3. Methods of characterization

2.3.1. Scanning Electron Microscope tests

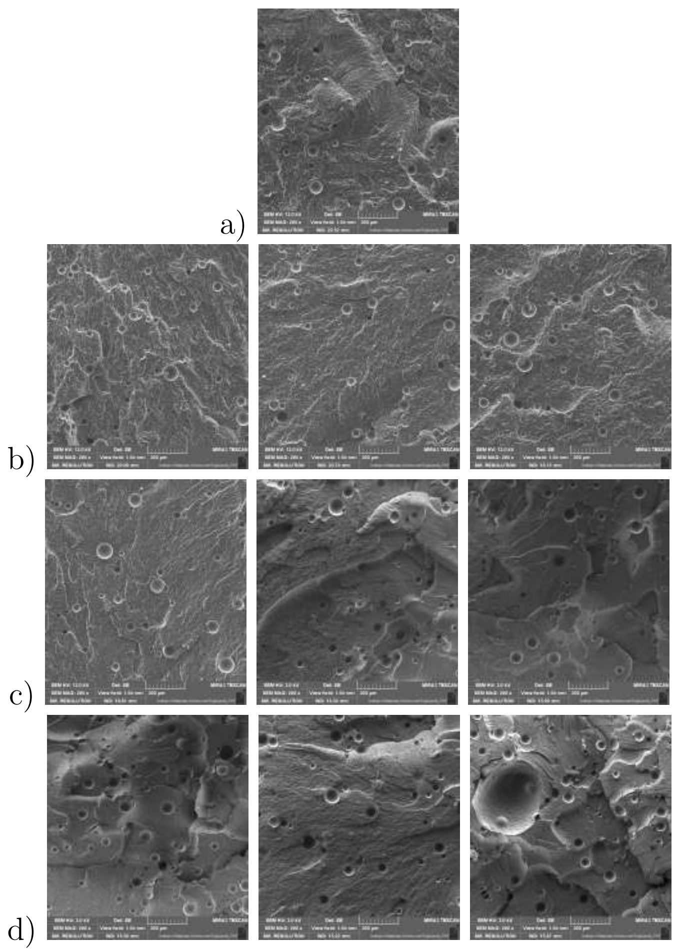


Figure 2: Fracture structure at 200 times magnification of a) the reference sample, and samples after conditioning at temperatures of b) 40°C for 7, 14, and 28 days, c) 60°C for 7, 14, and 28 days, d) 80°C for 7, 14, and 28 days for DP490 adhesive, respectively

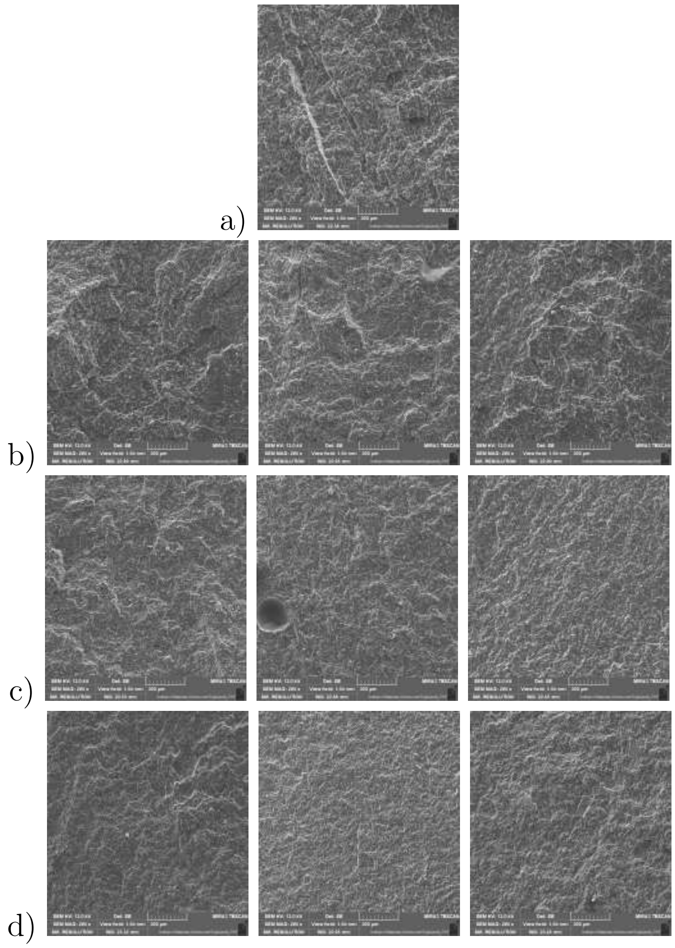


Figure 3: Fracture structure at 200 times magnification of: a) the reference sample, and samples after conditioning at temperatures of b) 40°C for 7, 14, and 28 days, c) 60°C for 7, 14, and 28 days, d) 80°C for 7, 14, and 28 days for DP125 Gray adhesive, respectively

Observation using a Scanning Electron Microscope MIRA3 TESCAN equipped with a Energy Dispersive Spectroscope (EDS, Ultim Max 65) was performed on both reference specimens and those exposed to conditioning actions. The samples were tested with an accelerating voltage of 12 kV and a working distance of 23 mm. The observations were performed in the contrast of secondary electrons (SE). A thin carbon coating of approximately 20 nm thickness was deposited on the samples using the Jeol JEE 4B vacuum evaporator to protect the samples from charging. A beam intensity (BI) of 14 and a dead time (DT) of 15% were used in the EDS analysis. The study aimed to evaluate the chemical composition of the adhesives and determine qualitative changes in the adhesive structure under the influence of conditioning on the fracture surfaces of the tested samples.

First, the chemical composition of the adhesives under test was analyzed. The DP490 adhesive primarily contains carbon (C), oxygen (O), silicon (Si), calcium (Ca), and titanium (Ti), along with minor quantities of sodium (Na), chlorine (Cl), and aluminum (Al). The DP125 Gray adhesive, in contrast, consists of carbon (C), oxygen (O), silicon (Si), aluminum (Al), fluorine (F), chlorine (Cl), sulfur (S), calcium (Ca), along with minor quantities of titanium (Ti) and iron (Fe).

As one can see DP125 Gray adhesive unfortunately contains fluorine (F). During normal use of adhesives containing fluorine compounds, the risk of toxicity is minimal. However, exposure to dust or fumes from burning or cutting materials containing fluorine should be avoided, as toxic decomposition products may be formed. Unfortunately, both adhesives also contain chlorine (Cl) and aluminum (Al), which are toxic to aquatic organisms. Additionally, DP490 contains sodium (Na), which can also be harmful to aquatic life.

Second, microscopic images of the fracture surface at 200 times magnification were analyzed for both adhesives (Figs 2, 3). In the first step, reference samples were compared with samples exposed to elevated temperatures. Conditioning the DP490 adhesive induces visible changes in the microstructure of the surface, which undoubtedly affects its mechanical and strength properties (Fig. 2). The fracture surface of the reference sample for DP490 adhesive has a rough, relatively flat surface with jagged edges, indicating a sudden failure with minimal plastic deformation. On the other hand, the fracture surface of the DP490 adhesive after conditioning is smoother and has a fibrous appearance, indicating a higher proportion of plastic deformation. The DP490 adhesive also shows hollow circular spaces, indicating the presence of air micropores in the adhesive structure. Pores vary in size and are randomly distributed, indicating their irregular occurrence in the adhesive. The presence of pores can affect the mechanical strength of the adhesive. Pores can act as stress concentrators, leading to the initiation of cracks and weakening the adhesive. The presence of micropores is due to the nature of the DP490 adhesive, and their formation cannot be avoided during adhesive application [33]. According to the authors opinion, this problem likely affects most thixotropic adhesives due to their inferior spreadability. Furthermore, for the DP490 adhesive, a clear change in the structure of the fracture surfaces are observable from an conditioning temperature of 60°C after just 14 days

(Fig. 2). This change continues over 28 days and is further evident at 80°C from 7 to 28 days. Importantly the structural change is clearly correlated with the tensile strength of the adhesive (Fig. 5b), which decreases rapidly by 23% relative to the reference value (31.14 MPa) at 60°C during the first 14 days, and next remains relatively constant.
In the case of the DP125 Gray adhesive, the differences between the fracture surfaces are not as pronounced (Fig. 3). The fracture surfaces of the DP125 Gray adhesive samples also indicates rather brittle fracture, regardless of conditioning at elevated temperatures. Unlike DP490 adhesive, DP125 Gray samples have no microporous air inclusions.
For the other conditioning methods, namely exposure to hygrothermal or freezing and thawing conditions, there were no significant differences in the fracture structures of the samples. Additionally, no changes were observed in the chemical composition of the adhesives.

2.3.2. Tensile tests

Tensile tests were performed according to EN ISO 527-1:2019, EN ISO 527-2:2012 standards. Epoxy samples (Fig. 4) were examined using a Zwick Z100 universal testing machine under displacement control at a constant rate of 1 mm/min. A load cell, capable of handling a maximum load of 5 kN, was used to measure the applied force. Contact extensometer (Zwick BZ2-EXED550P - measuring base 15 mm) and strain gauges (HBM 1-LY11-

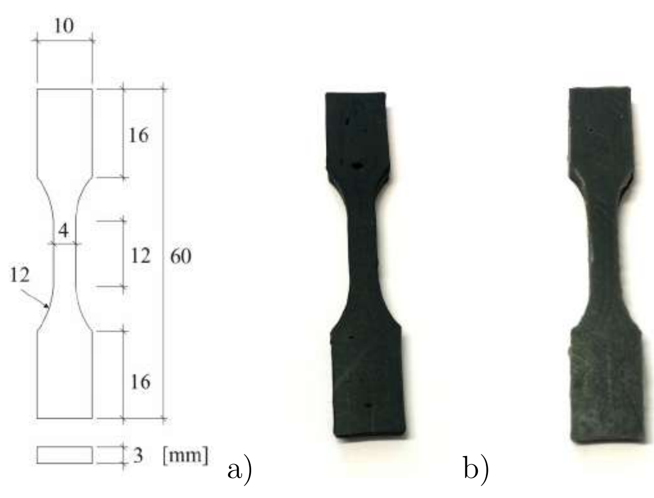
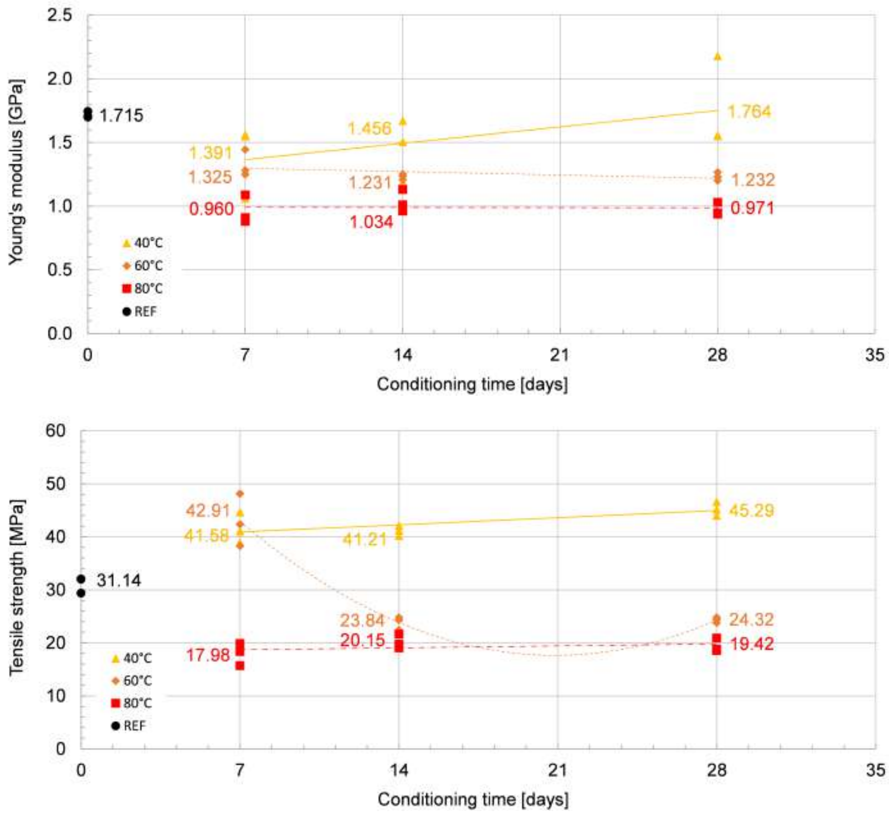


Figure 4: Specimen's geometry: a) DP490, b) DP125 Gray

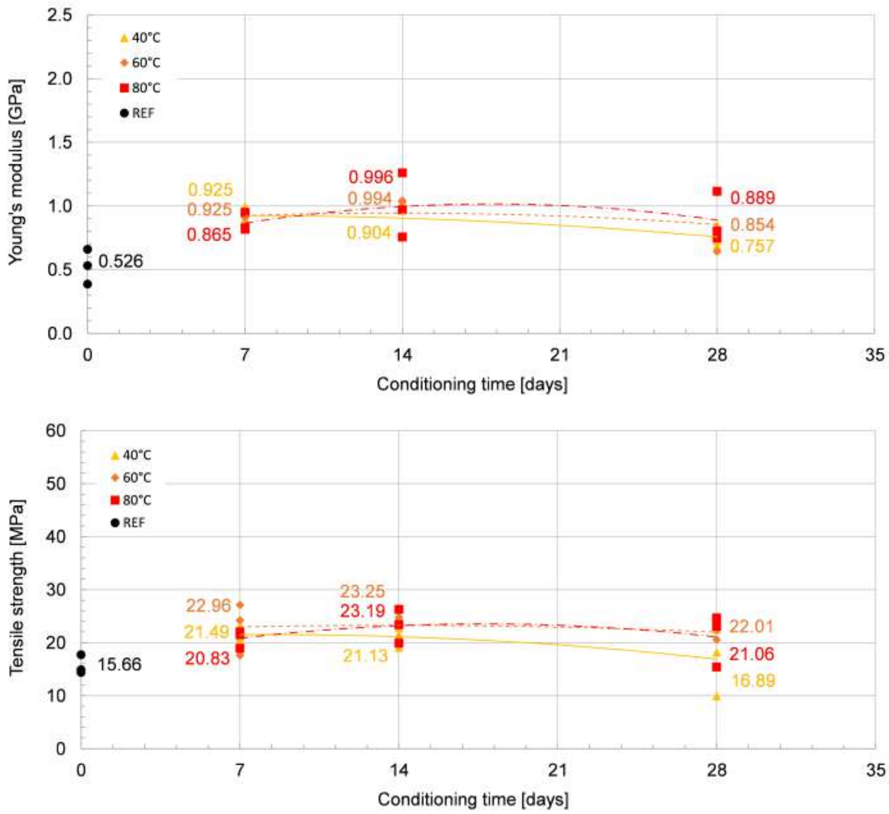


a)

b)

Figure 5: The variation in a) Young's modulus and b) tensile strength of DP490 adhesive over time at temperatures of 40°C, 60°C, and 80°C, compared to reference values

0.6/120 + HBM universal amplifier 1-MX840B) were used to determine strains. Before conducting the tests, the dimensions of each specimen—thickness and width—were precisely measured in three separate sections using digital calipers with 0.01 mm accuracy. These measurements were used to compute the average cross-sectional area, essential for assessing the longitudinal normal stress. The modulus E was calculated from the slope of the secant line connecting the 0.1% and 0.3% strain points on the stress-strain curve. The average Young's moduli derived from the reference samples were 1.715 GPa and 0.526 GPa for DP490 and DP125 Gray, respectively. Poisson's ratio ν was determined by calculating the slope of the linear trend line using experimental strain data in both directions, up to 0.2% axial strain. The average Poisson's ratios derived from the reference samples were 0.37 and 0.48 for



a)

b)

Figure 6: The variation in a) Young's modulus and b) tensile strength of DP125 Gray adhesive over time at temperatures of 40°C, 60°C, and 80°C, compared to reference values

DP490 and DP125 Gray, respectively. Poisson's ratios were determined from the quotient of the strain in the direction perpendicular to the applied force (ε_p) to the strain in the direction of the applied force (ε_n) over multiple loading cycles for each reference sample tested ($\nu = -\varepsilon_p/\varepsilon_n$). All the coefficients of determination (R^2) for each fitted curve were computed, resulting in an average of 0.99. This average suggests that the experimental curve remains almost linear across the assessed range of strain values.

The tensile strengths of the tested reference samples were 31.14 MPa (at 23°C, 55%RH) for the DP490 adhesive and 15.66 MPa (at 23°C, 55%RH) for the DP125 Gray adhesive. The obtained average tensile strengths from our tests were lower than the values quoted by the manufacturer: for the DP490 adhesive by 13.5% (the manufacturer's reference value is 36.00 MPa), and for the DP125 Gray adhesive by up to 31.2% (the manufacturer's reference value is 22.75 MPa). Additionally, Fig. 1 compares the tensile strength and Young's modulus values obtained from the tests for the DP490 and DP125 Gray adhesives with the parameters of other epoxy adhesives reported in the literature.

2.3.3. Tensile tests - effects of elevated temperature conditioning

Figures 5 and 6 show the effect elevated temperature conditioning at 40°C, 60°C, and 80°C for 7, 14, and 28 days on the Young's modulus (Figs 5a, 6a) and tensile strength (Figs 5b, 6b) of DP490 and DP125 Gray adhesives, respectively, compared to reference values. As can be seen, the behavior of the two tested adhesives under conditioning at elevated temperatures is quite different.

The elastic modulus of the DP490 adhesive (Fig. 5a) decreases as the conditioning temperature increases. The higher the temperature, the greater the decrease in the Young's modulus. The largest change in Young's modulus occurred at a conditioning temperature of 80°C for 7 days, amounting to a 44% decrease from the average reference value (1.715 GPa), resulting in an elastic modulus of 0.960 GPa. When conditioning at 40°C, it initially decreases relative to the reference value by 18.9% but increases by 2.8% relative to the reference value over time (28 days). In the case of conditioning at 60°C, the value of Young's modulus successively decreases from the very beginning over time. During conditioning at 80°C, the value of Young's modulus initially decreases and then remains constant over time.

In the case of the DP490 adhesive, the change in tensile strength values (Fig. 5b) relative to the average reference value (31.14 MPa) at 40°C and 60°C during 7 days of conditioning increases quite significantly, by a maximum of 27.4% at 60°C. Over time, at 60°C, it decreases relative to the reference value by 21.9%, while at 40°C, it continues to increase over time until it exceeds the reference value by 31.2%. At a conditioning temperature of 80°C, the tensile strength decreases by 42.3% from the reference value after just 7 days and remains constant over time, up to 28 days.

In conclusion, if one wants to increase the tensile strength of DP490 adhesive and improve its elasticity at the same time, it is necessary to anneal it at 40°C for 28 days. Under these conditions, the elastic modulus of DP490 adhesive will increase slightly, by approximately 2.5%, and, most importantly, it will not decrease. Additionally, the tensile strength will increase by about 31.2%.

For the DP125 Gray adhesive, the overall behavior of the Young's mod-

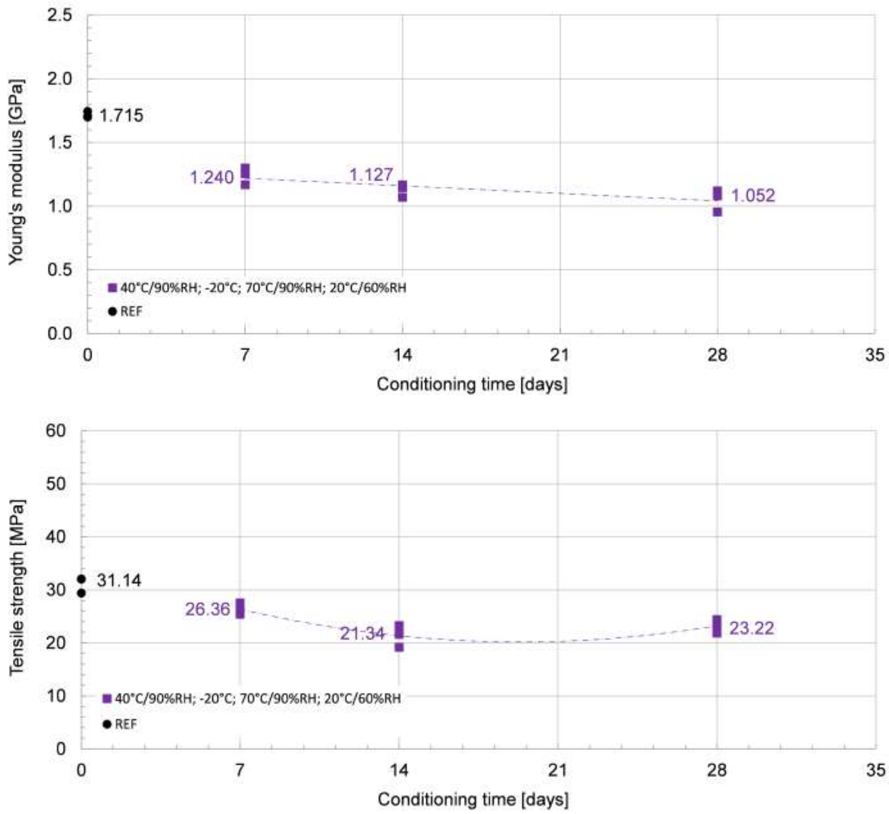


Figure 7: The variation in a) Young's modulus and b) tensile strength of DP490 adhesive over time due to hygrothermal conditioning, compared to reference values

ulus values (Fig. 6a) is different from that of the DP490 adhesive. This is because the all values of the elastic modulus are increasing relative to the average reference value (0.526 GPa). The largest average increase of 47.2% in Young's modulus values was recorded when the samples were conditioned at 80°C for 14 days. However, after 14 days at a conditioning temperature of 80°C, the Young's modulus begins to decrease. For lower conditioning temperatures, i.e., 40°C and 60°C, the elasticity moduli begin to decrease after just 7 days. The lowest value of Young's modulus obtained in the study was 12.5% higher than the reference value, at 0.757 GPa (the sample was conditioned at 40°C for 28 days).

The situation is very similar for tensile strength (Fig. 6b). The highest tensile strength value is approximately 23.00 MPa, which is 32.2% higher than

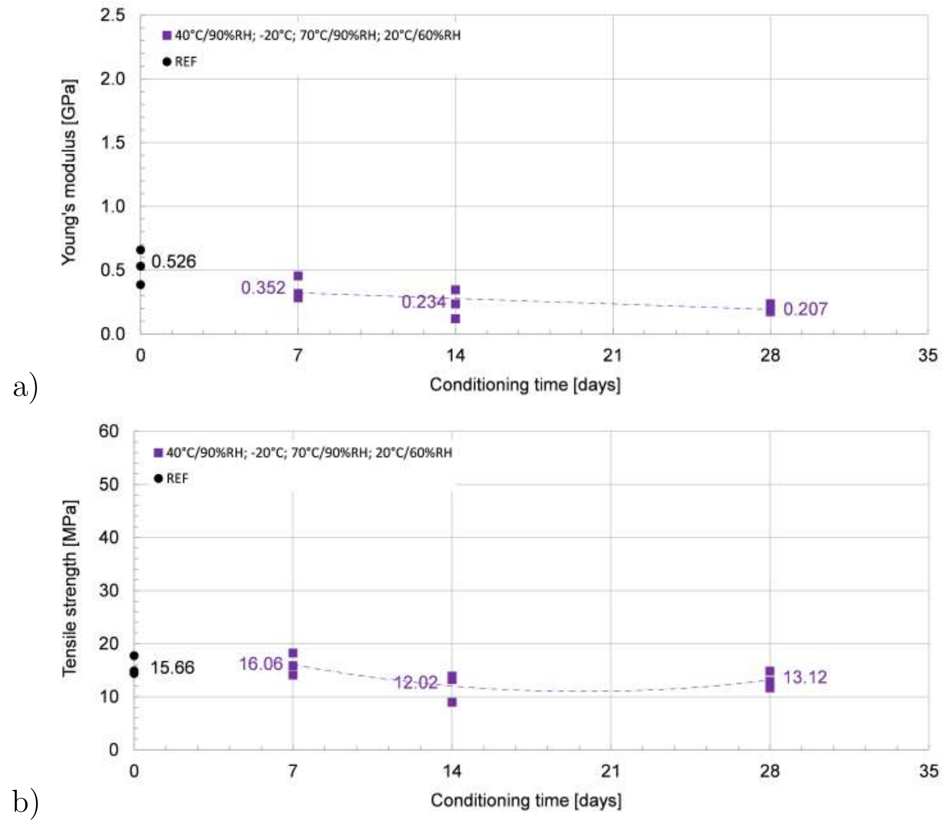
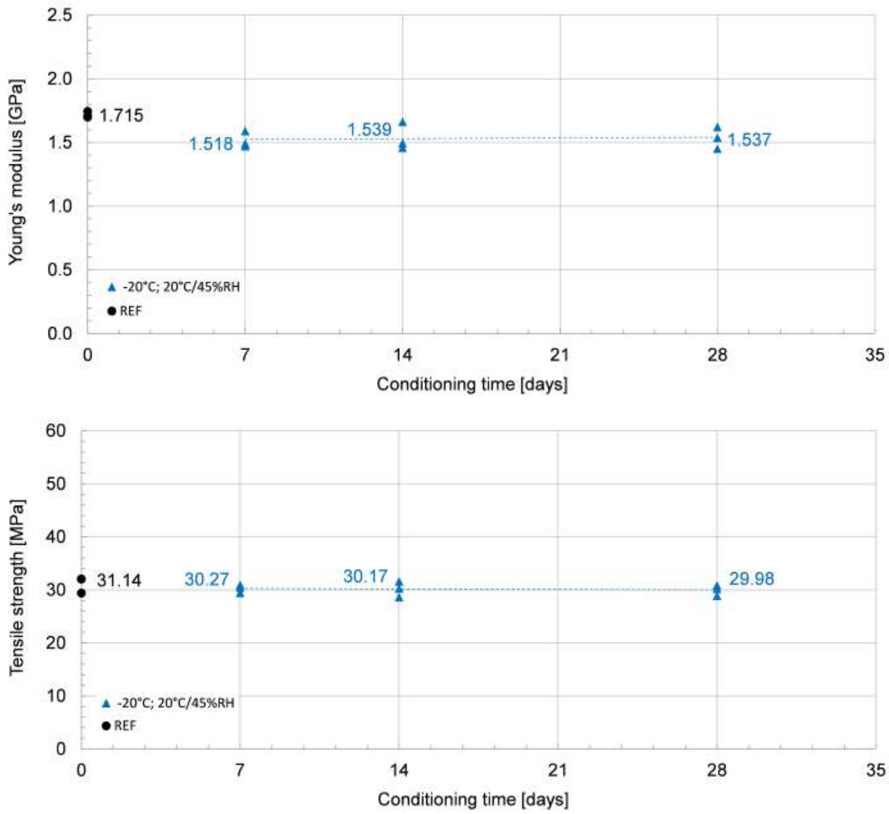


Figure 8: The variation in a) Young's modulus and b) tensile strength of DP125 Gray adhesive over time due to hygrothermal conditioning, compared to reference values

the reference value (15.66 MPa). The maximum value is associated with conditioning at 60°C and 80°C for 14 days. After 14 days at conditioning temperatures of 60°C and 80°C, the tensile strength begins to decrease but is still about 25% higher after 28 days than the reference tensile strength value. At a conditioning temperature of 40°C, the maximum tensile strength is reached after 7 days, and over time, this strength drops to a level close to the reference value.

In conclusion, in the case of DP125 Gray adhesive, the highest increase in the values of modulus of elasticity (about 47.2%) and tensile strength (about 32.6%) relative to the reference values will be obtained when the adhesive is annealed at 60°C to 80°C for 14 days.

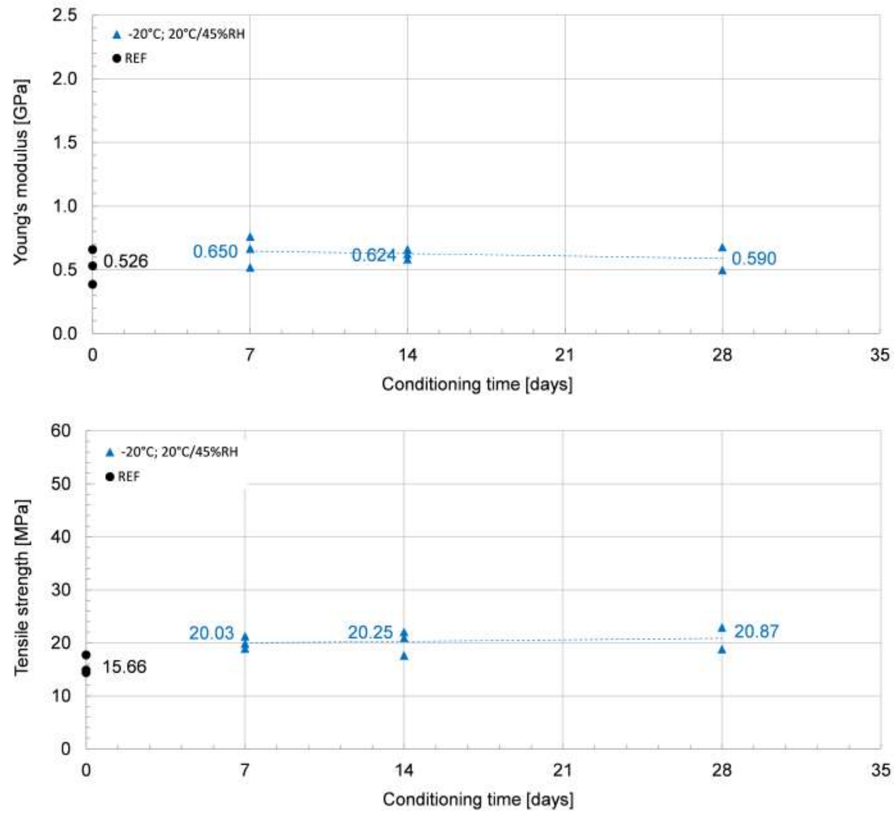


a) b)

Figure 9: The variation in a) Young's modulus and b) tensile strength of the DP490 adhesive over time due to freeze-thaw conditioning, compared to reference values

2.3.4. Tensile tests - effects of hygrothermal conditioning

Figures 7 and 8 show how the Young's modulus and tensile strength values change for the tested adhesives due to hygrothermal conditioning. In the case of DP490 adhesive (Fig. 7a), hygrothermal conditioning results in a steady 38.6% decrease in Young's modulus compared to the reference value (1.715 GPa) over a period of up to 28 days. Tensile strength (Fig. 7b), on the other hand, decreases by 31.5% relative to the reference value (31.14 MPa) by day 14 and then increases slightly to 23.22 MPa by day 28. In the case of DP125 Gray adhesive, there is a significant decrease in Young's modulus over time, with a total reduction of up to 60% relative to the reference value (0.526 GPa) after 28 days (Fig. 8a). In the case of this adhesive, the tensile strength initially increases slightly, decreases by 23.2% after 14 days of conditioning,



a)

b)

Figure 10: The variation in a) Young's modulus and b) tensile strength of the DP125 Gray adhesive over time due to freeze-thaw conditioning, compared to reference values

and then increases again after 28 days (Fig. 8b).

2.3.5. Tensile tests - effects of freeze-thaw conditioning

Figures 9 and 10 show how the Young's modulus and tensile strength values change for the tested adhesives due to freeze-thaw conditioning. With DP490 adhesive (Fig. 9a), Young's modulus decreases by approximately 11.5% and remains constant over 28 days. For the same adhesive, the tensile strength (Fig. 9b) also decreases slightly during the conditioning process, by up to 3.7%. In the case of DP125 Gray adhesive, both Young's modulus and tensile strength values increase during conditioning and remain rather stable over time. For Young's modulus, there is an initial increase of 19% from the reference value, while tensile strength shows an increase of 21.8%.

2.3.6. Shore hardness tests

The analysis also assessed the hardness parameter of the tested adhesives. The study was conducted using Shore's method. The test procedures were standardized in compliance with ISO 48-4 and ASTM D2240 type D scales. Shore's reference hardness values for the tested adhesives are 73.6 SH and 65.2 SH for DP490 and DP125 Gray, respectively. Shore hardness will be used for additional evaluation of changes in the mechanical properties of the material over time under thermal, hygrothermal, and freeze-thaw conditions. This is because a decrease in hardness is typically associated with material degradation over time.

2.3.7. Shore hardness tests - effects of elevated temperature conditioning

Figure 11 shows the effect of elevated temperature conditioning at 40°C, 60°C, and 80°C for 7, 14, and 28 days on the Shore hardness of DP490 and DP125 Gray adhesives compared to reference values. In the case of DP490 adhesive, the Shore hardness value stays rather consistent with the reference Shore hardness value (73.6 SH) throughout the elevated temperature conditioning process (Fig. 11a). In contrast to DP490 adhesive, the Shore hardness value of DP125 Gray adhesive changes noticeably during the conditioning process at elevated temperatures, by as much as 10% on average (Fig. 11b). An increase in hardness of 10% relative to the reference value (65.2 SH) already occurs at a conditioning temperature of 40°C during 7 days, and thereafter the hardness values tend not to change significantly.

2.3.8. Shore hardness tests - effects of hygrothermal conditioning

Figure 12 shows the effect of hygrothermal conditioning at 7, 14, and 28 days on Shore hardness compared to reference samples. For the DP490 adhesive (Fig. 12a), the Shore hardness value progressively decreases over time, reaching a maximum reduction of approximately 7% relative to the reference value (73.6 SH) at 28 days. Similarly, for the DP125 Gray adhesive (Fig. 12b), there is a decrease in Shore hardness, with a maximum reduction of 4.7% relative to the reference value (65.2 SH) within 28 days.

2.3.9. Shore hardness tests - effects of freeze-thaw conditioning

Figure 13 shows the effect of freezing and thawing on DP490 and DP125 Gray adhesives at 7, 14, and 28 days. As can be seen, the process of repeated freezing and thawing does not significantly change the Shore hardness of the DP490 adhesive relative to the reference values, while that of the DP125

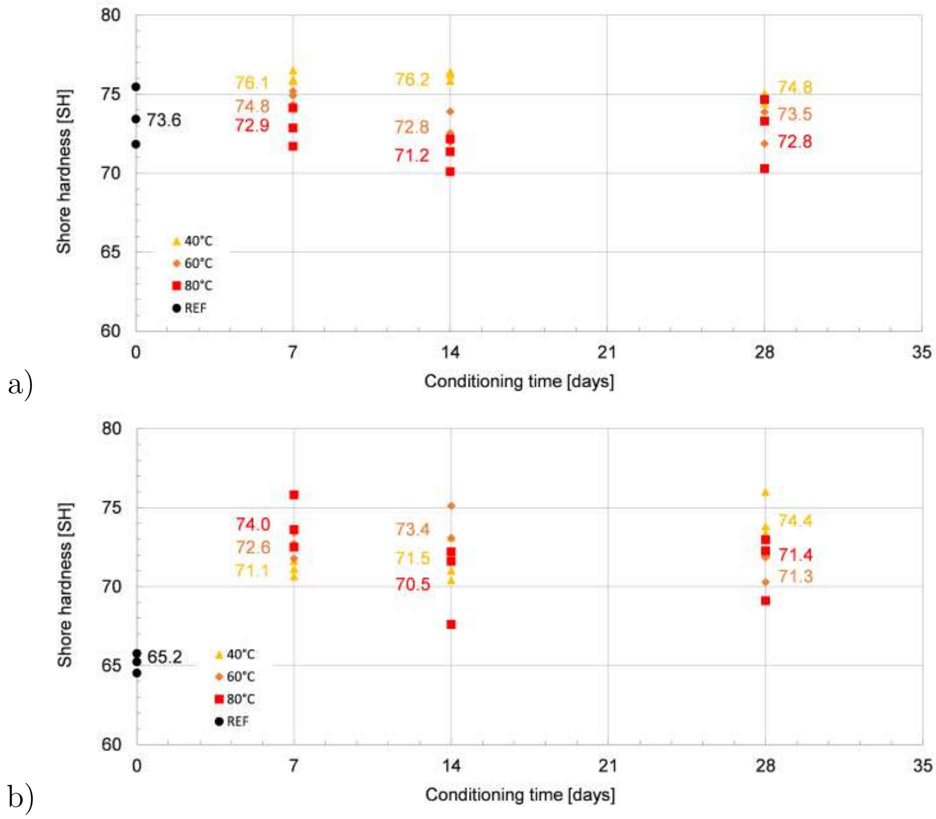


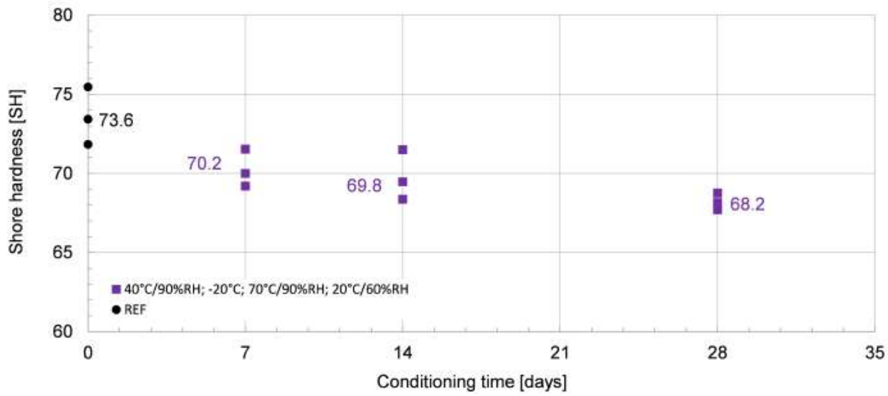
Figure 11: The variation in Shore hardness of a) DP490 and b) DP125 Gray adhesives over time at temperatures of 40°C, 60°C, and 80°C, compared to reference values

Gray adhesive initially increases by about 6.9% over the reference value until day 14, and then decreases.

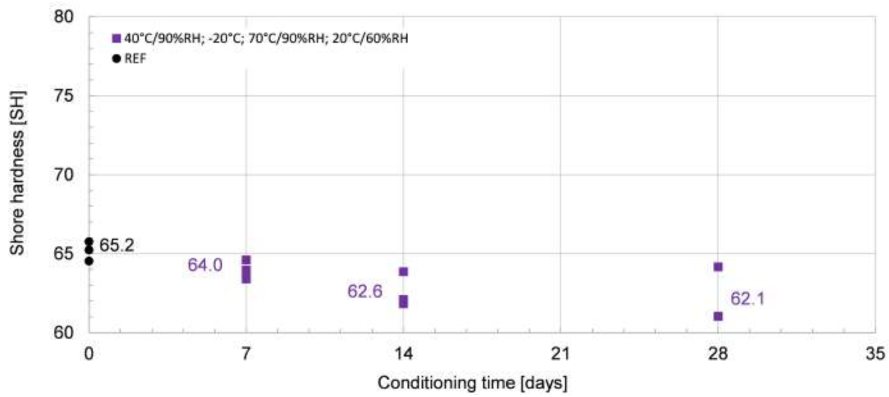
3. Conclusions

In this study, the impact of various environmental conditions on commercial epoxy adhesives employed in structural applications was examined and evaluated. Based on the results obtained, the following key conclusions can be drawn:

- Observations using a scanning electron microscope (SEM) revealed that, unfortunately, one of the adhesives, specifically DP125 Gray, contains fluorine—an element not naturally found in the environment and



a)



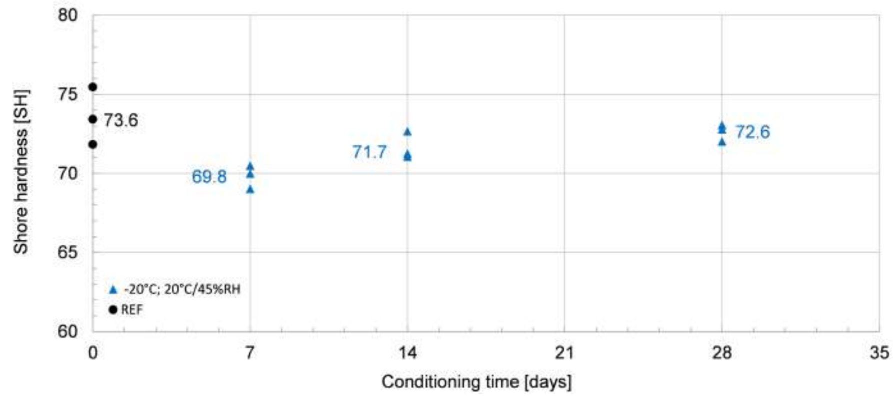
b)

Figure 12: The variation in Shore hardness of a) DP490 and b) DP125 Gray adhesives over time under the effects of hygrothermal conditioning, compared to reference values

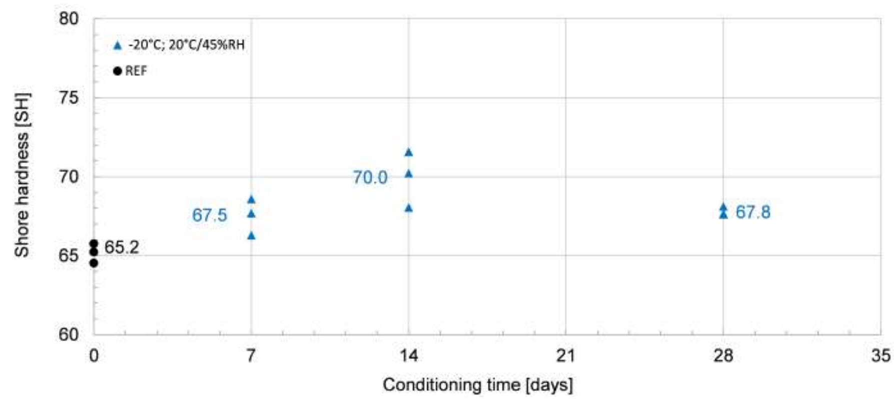
classified as harmful by the Stockholm Convention on Persistent Organic Pollutants.

For both adhesives tested, the samples exposed to environmental conditions showed no change in their chemical composition compared to the reference samples.

Additionally, tests conducted on the DP490 adhesive using SEM revealed distinct changes in the fracture structures of samples exposed to high temperatures (from 60°C for 14 days), despite the absence of significant changes in the Shore hardness parameters. No changes in the fracture structures were observed with the other conditioning methods for any of the adhesives.



a)



b)

Figure 13: The variation in Shore hardness of a) DP490 and b) DP125 Gray adhesives over time under the effects of freeze-thaw conditioning, compared to reference values

Furthermore, it should be noted that the DP490 adhesive consistently contains micropores, which could undoubtedly affect the load-bearing capacity of connections formed with this adhesive.

- In the case of the DP490 adhesive, conditioning the samples at elevated temperatures resulted in a significant decrease in the material's elasticity, particularly at higher temperatures. However, at lower annealing temperatures, such as 40°C, an initial decrease in elasticity is observed, followed by an increase above the reference values after 28 days. Long-term exposure to a temperature of 40°C also leads to an increase in tensile strength over time, whereas at higher temperatures, such as 80°C, there is a sharp decrease in tensile strength. In addition, it should be noted that in the case of DP490 adhesive, exposure to elevated temperatures does not significantly change the Shore hardness parameter.

In the case of DP125 Gray adhesive, thermal conditioning of the samples at elevated temperatures resulted in an increase in both Young's modulus and tensile strength. In addition, it should be noted that in the case of DP125 Gray adhesive, exposure to elevated temperatures, almost regardless of the specific temperature and duration, significantly increases the Shore hardness parameter. Thus, a correlation can be observed between the Shore hardness and the values of Young's modulus and tensile strength; as the hardness increases, the elasticity and strength values also increase.

- When the samples were conditioned under varying temperatures and increased humidity, there was a significant decrease in the material's elasticity, tensile strength, and Shore hardness. The primary reason for this was likely the high humidity, which caused the adhesives to attract water molecules. Therefore, in practical applications of these epoxy structural adhesives, special care must be taken to prevent exposure to water or high humidity. It may also be advisable to apply additional external impregnation to such connections to limit water access.

- When both adhesives were conditioned by freezing and thawing the samples, the process did not have a significant impact on the elasticity parameter, tensile strength, or Shore hardness. In the case of the DP490 adhesive, slight decreases in these values can be observed, while in the case of DP125 Gray, these values even show a slight increase. In real weather conditions, a temporary drop in temperature to -20°C will therefore have minimal impact on the behavior of the adhesives.

Acknowledgements

Financial support from the National Science Centre, Poland, under grant 2023/07/X/ST8/00497 titled "Failure analysis in a single-lap adhesive joint of thin sheets" within the "MINIATURA 7" program is gratefully acknowledged.

References

[1] H. Pham, M. Marks, Epoxy resins, Wiley, 2005.

[2] Z. Ahmadi, Nanostructured epoxy adhesives: A review, Progress in Organic Coatings 135 (2019) 449–453.

[3] R. Bagheri, B. Marouf, R. Pearson, Rubber-toughened epoxies: A critical review, Journal of Macromolecular Science, Part C: Polymer Reviews 49 (2009) 201–225.

[4] P. Kumar, A. Patnaik, S. Chaudhary, A review on application of structural adhesives in concrete and steel–concrete composite and factors influencing the performance of composite connections, International Journal of Adhesion and Adhesives 77 (2017) 1–14.

[5] B. Täljsten, The importance of bonding: an historic overview and future possibilities, in: International Symposium on Bond Behaviour of FRP in Structures: 07/12/2005-09/12/2005, International Institute for FRP in Construction, 2005, pp. 1–10.

[6] R. Carbas, E. Marques, L. da Silva, A. Lopes, Effect of cure temperature on the glass transition temperature and mechanical properties of epoxy adhesives, The Journal of Adhesion 90 (2014) 104–119.

[7] P. Silva, P. Fernandes, J. Sena-Cruz, J. Xavier, F. Castro, D. S. V. Carneiro, Effects of different environmental conditions on the mechanical characteristics of a structural epoxy, Composites Part B 88 (2016) 55–63.

[8] J. Michels, R. Widmann, C. Czaderski, R. Allahvirdizadeh, M. Motavalli, Glass transition evaluation of commercially available epoxy resins used for civil engineering applications, Composites Part B 77 (2015) 484–493.

[9] Y. Lin, X. Chen, Moisture sorption–desorption–resorption characteristics and its effect on the mechanical behavior of the epoxy system, Polymer 46 (2005) 11994–12003.

[10] Q. Yang, G. Xian, V. Karbhari, Hygrothermal ageing of an epoxy adhesive used in FRP strengthening of concrete, Journal of Applied Polymer Science 107 (2008) 2607–2617.

[11] M. Lettieri, M. Frigione, Effects of humid environment on thermal and mechanical properties of a cold-curing structural epoxy adhesive, Construction and Building Materials 30 (2012) 753–760.

[12] J. Yagoubi, G. Lubineau, S. Saghir, J. Verdu, A. Askari, Thermomechanical and hygroelastic properties of an epoxy system under humid and cold-warm cycling conditions, Polymer Degradation and Stability 99 (2014) 146–155.

[13] O. Oni, M. Lawrence, M. Zappi, W. Chirdon, A review of strategies to enhance the water resistance of green wood adhesives produced from sustainable protein sources, Sustainability 15 (2023) 14779.

[14] S. Akpinar, M. D. Aydin, A. Özel, A study on 3D stress distributions in the bi-adhesively bonded T-joints, Applied Mathematical Modelling 37 (24) (2013) 10220–10230.

[15] R. Carbas, E. Marques, L. Da Silva, A. Lopes, Effect of cure temperature on the glass transition temperature and mechanical properties of epoxy adhesives, The Journal of Adhesion 90 (1) (2014) 104–119.

[16] L. da Silva, R. Adams, Joint strength predictions for adhesive joints to be used over a wide temperature range, International Journal of Adhesion and Adhesives 27 (5) (2007) 362–379.

[17] M. Banea, L. da Silva, Adhesively bonded joints in composite materials: an overview, Proceedings of the Institution of Mechanical Engineers, Part L: Journal of Materials: Design and Applications 223 (1) (2009) 1–18.

[18] M. Banea, L. da Silva, R. Campilho, Effect of temperature on tensile strength and mode I fracture toughness of a high temperature epoxy adhesive, Journal of Adhesion Science and Technology 26 (7) (2012) 939–953.

[19] M. D. Banea, L. F. da Silva, R. Campilho, Effect of temperature on the shear strength of aluminium single lap bonded joints for high temperature applications, Journal of Adhesion Science and Technology 28 (14-15) (2014) 1367–1381.

[20] J. Firmo, M. Roquette, J. Correia, A. Azevedo, Influence of elevated temperatures on epoxy adhesive used in CFRP strengthening systems for civil engineering applications, International Journal of Adhesion and Adhesives 93 (2019) 102333.

[21] R. Campilho, A. Pinto, M. D. Banea, R. Silva, L. F. da Silva, Strength improvement of adhesively-bonded joints using a reverse-bent geometry, Journal of Adhesion Science and Technology 25 (18) (2011) 2351–2368.

[22] B. de Oliveira, R. Campilho, F. da Silva, R. Rocha, Comparison between the ENF and 4ENF fracture characterization tests to evaluate G IIC of bonded aluminium joints, The Journal of Adhesion 94 (11) (2018) 910–931.

[23] Z. Chen, R. Adams, L. F. da Silva, Fracture toughness of bulk adhesives in mode I and mode III and curing effect, International Journal of Fracture 167 (2011) 221–234.

[24] J. da Costa, A. Akhavan-Safar, E. Marques, R. Carbas, L. da Silva, Cyclic ageing of adhesive materials, The Journal of Adhesion 98 (10) (2022) 1341–1357.

[25] I. Pires, L. Quintino, J. Durodola, A. Beevers, Performance of bi-adhesive bonded aluminium lap joints, International Journal of Adhesion and Adhesives 23 (3) (2003) 215–223.

[26] Ş. Temiz, Application of bi-adhesive in double-strap joints subjected to bending moment, Journal of Adhesion Science and Technology 20 (14) (2006) 1547–1560.

[27] S. Temiz, H. Adin, İ. Y. Sülü, Behaviour of bi-adhesive in double-strap joint with embedded patch subjected to bending, Journal of Theoretical and Applied Mechanics 45 (3) (2015) 83.

[28] H. Özer, Ö. Öz, A comparative evaluation of numerical and analytical solutions to the biadhesive single-lap joint, Mathematical Problems in Engineering 2014 (2014).

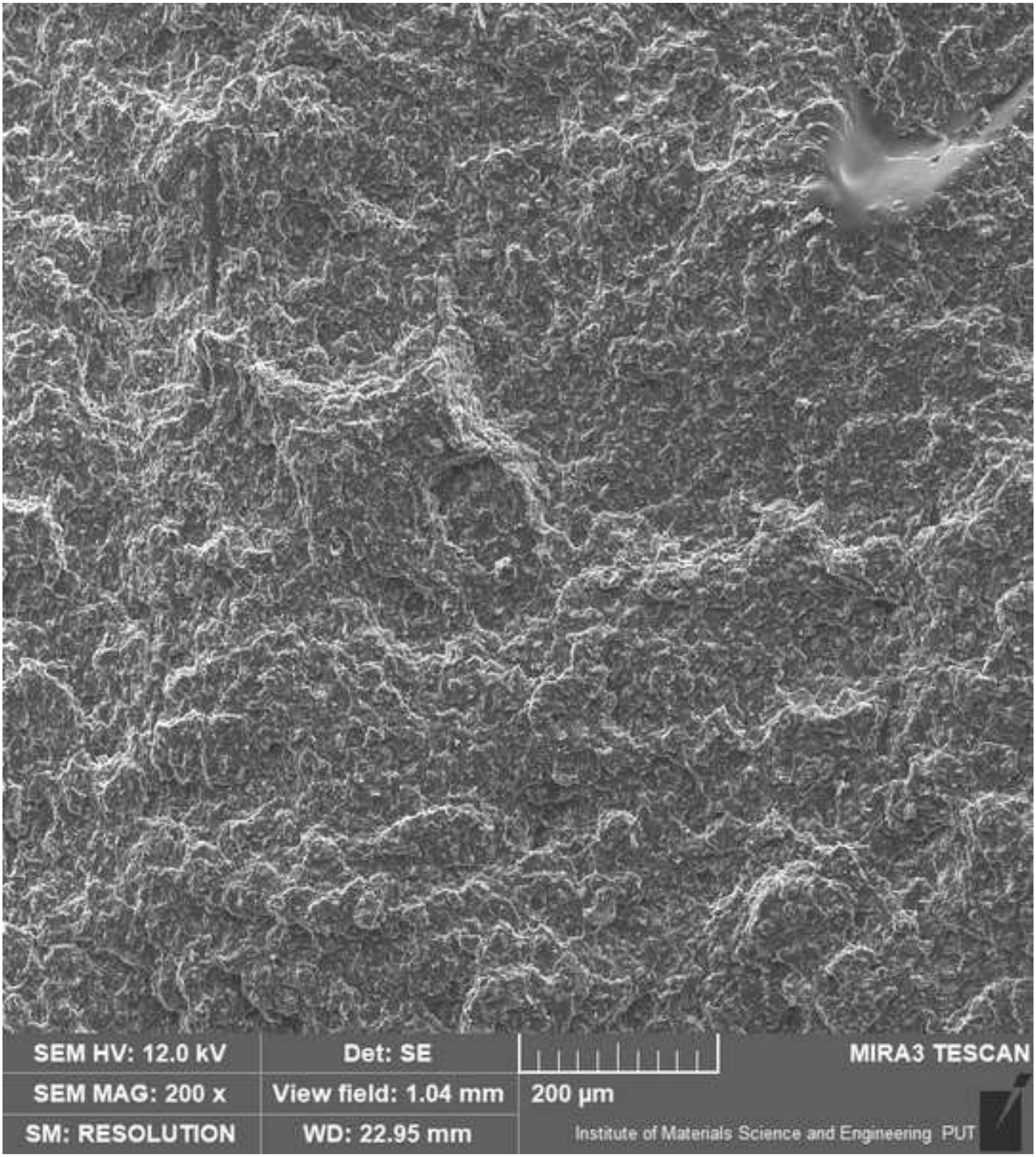
[29] P. Zdziebko, J. Korta, T. Uhl, High temperature impact assessment on the shear strength of adhesively bonded joints in multi-material design, Diagnostyka 16 (3) (2015) 9–15.

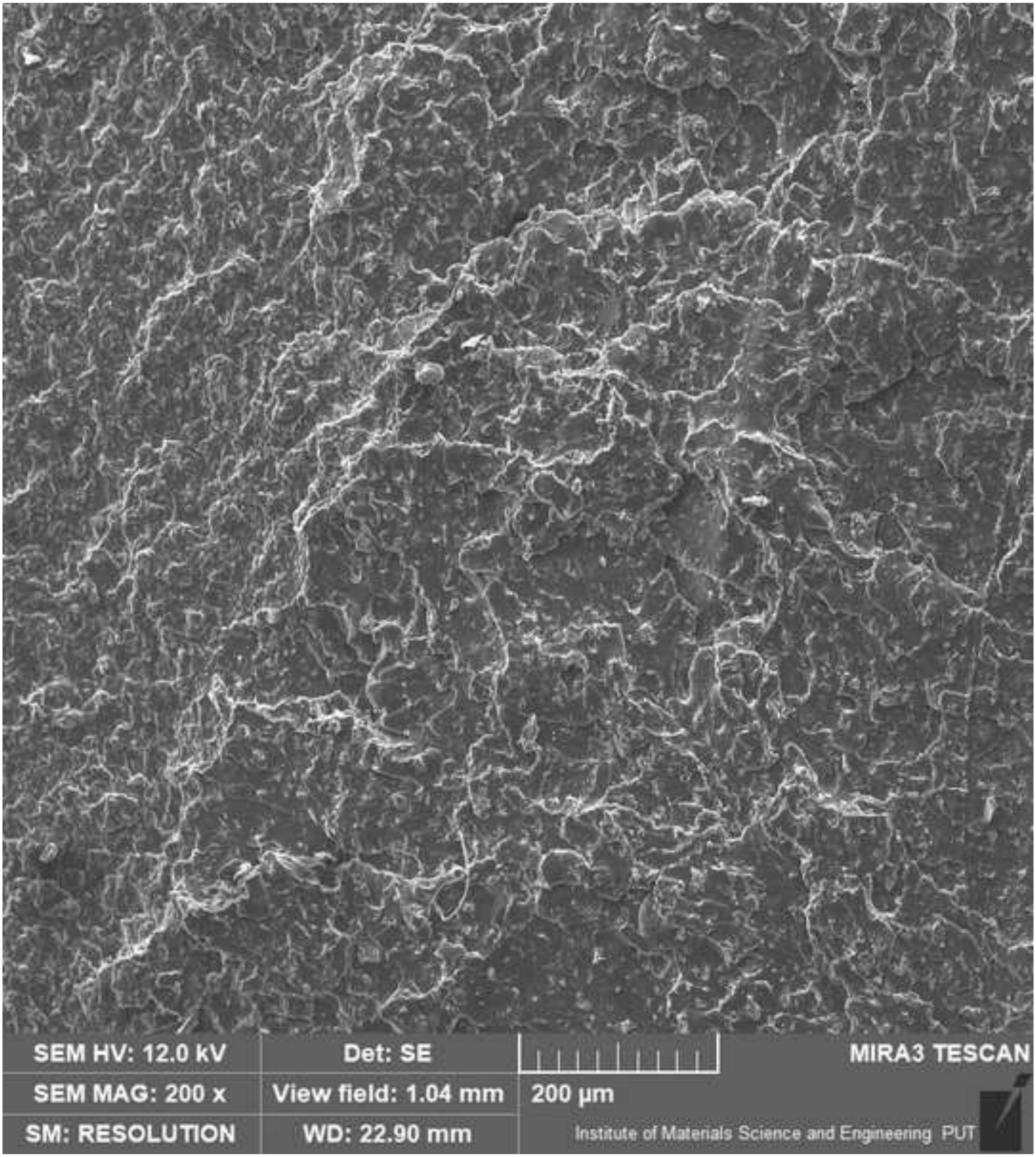
[30] S. Cabral-Fonseca, J. Correia, J. Custódio, H. Silva, A. Machado, J. Sousa, Durability of frp-concrete bonded joints in structural rehabilitation: A review, International Journal of Adhesion and Adhesives 83 (2018) 153–167.

[31] Ž. Unuk, A. Ivanič, V. Ž. Leskovar, M. Premrov, S. Lubej, Evaluation of a structural epoxy adhesive for timber-glass bonds under shear loading and different environmental conditions, International Journal of Adhesion and Adhesives 95 (2019) 102425.

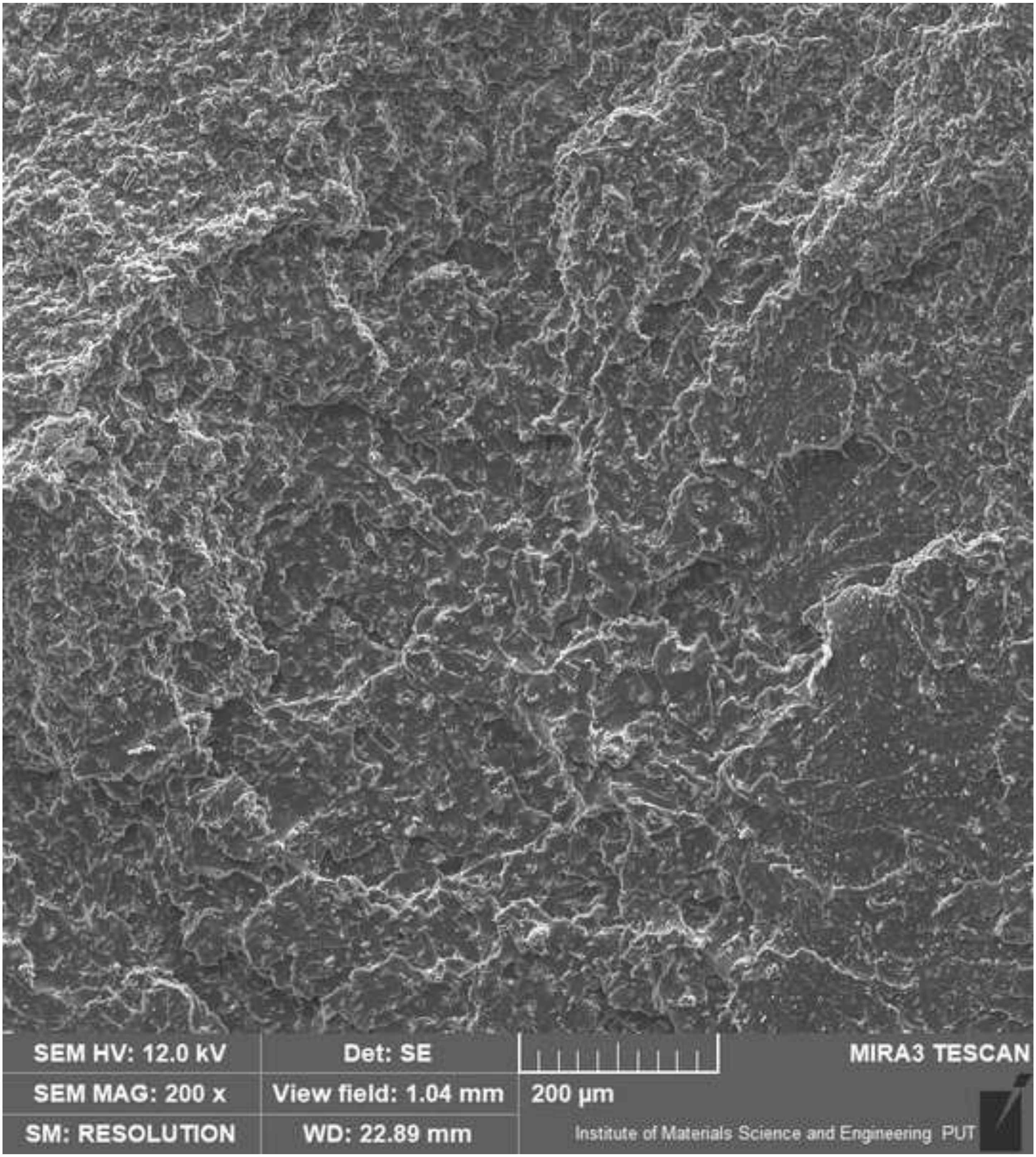
[32] F. Marchione, G. Chiappini, P. Munafò, Effect of temperature and relative humidity on the shear performance of double-lap adhesive joints between steel and glass adherends, Journal of Building Engineering 45 (2022) 103546.

[33] K. Winkelmann, F. Jan, Ł. Smakosz, V. Konopińska-Zmysłowska, V. Eremeyev, M. Kujawa, A random field-based simulational identification of possible levels of material imperfections of adhesive-bonded joints, Bulletin of the Polish Academy of Sciences: Technical Sciences 6 (2024) (Accepted for publication).

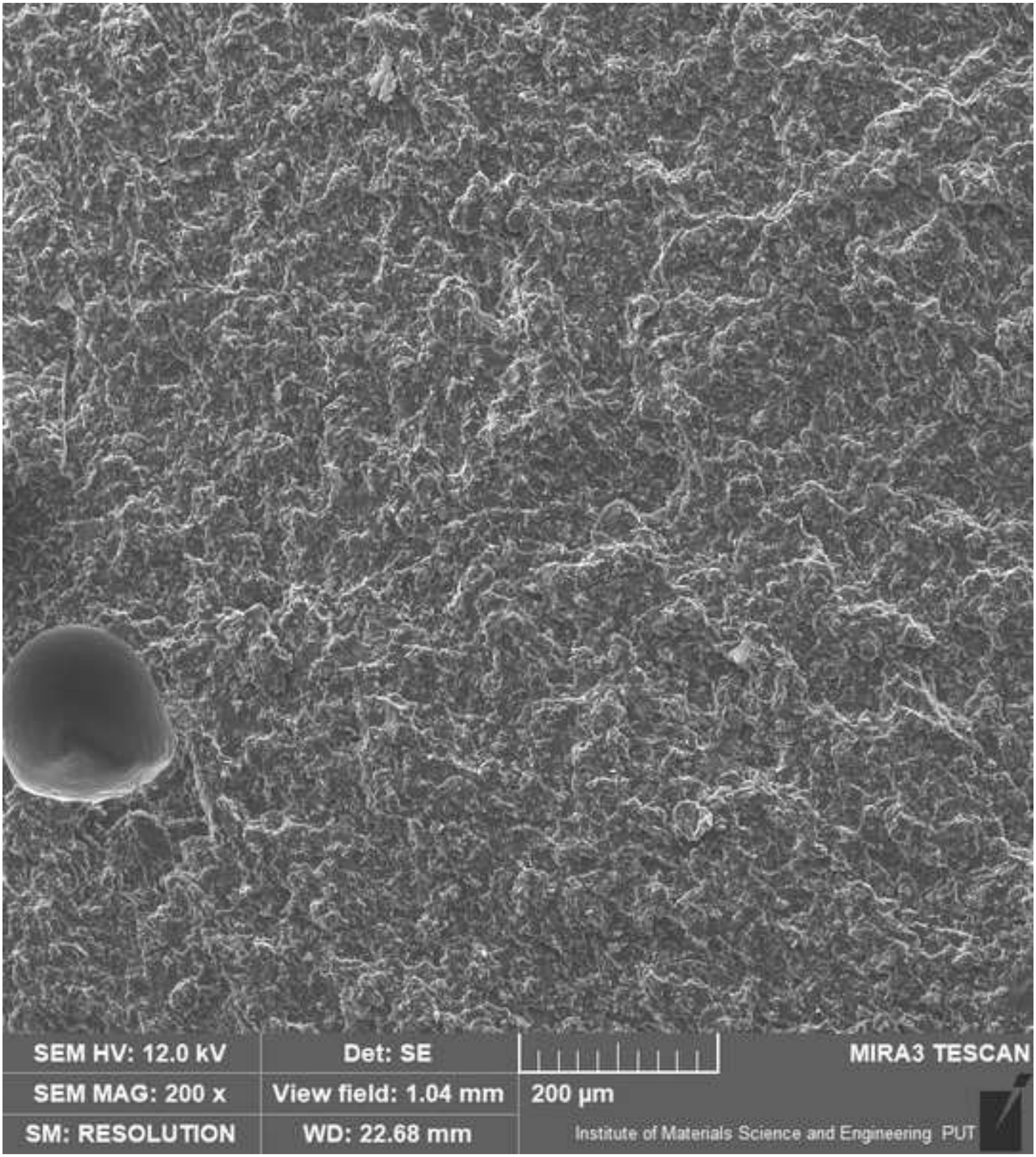

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 22.95 mm
Institute of Materials Science and Engineering PUT

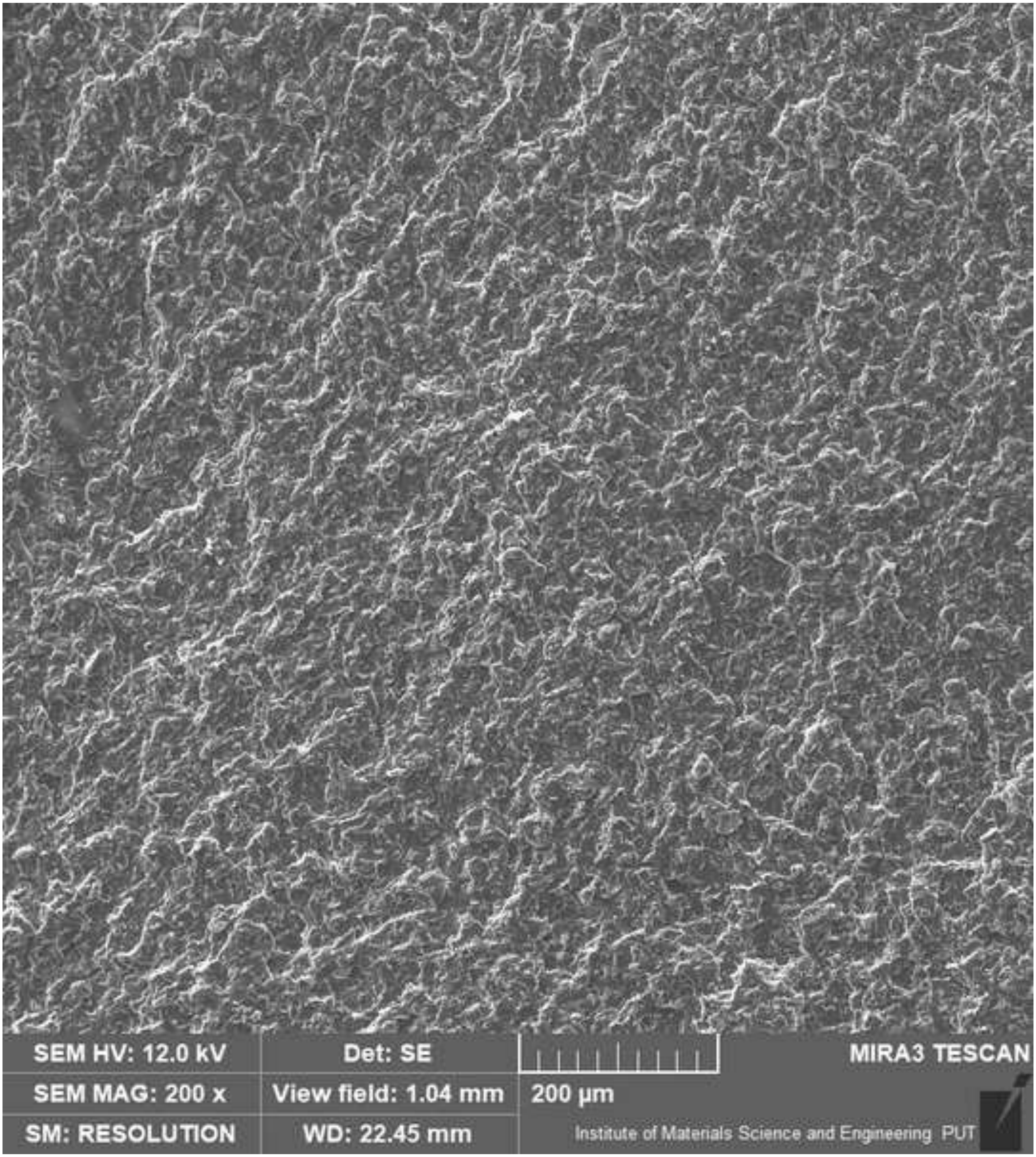

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 22.90 mm
Institute of Materials Science and Engineering PUT

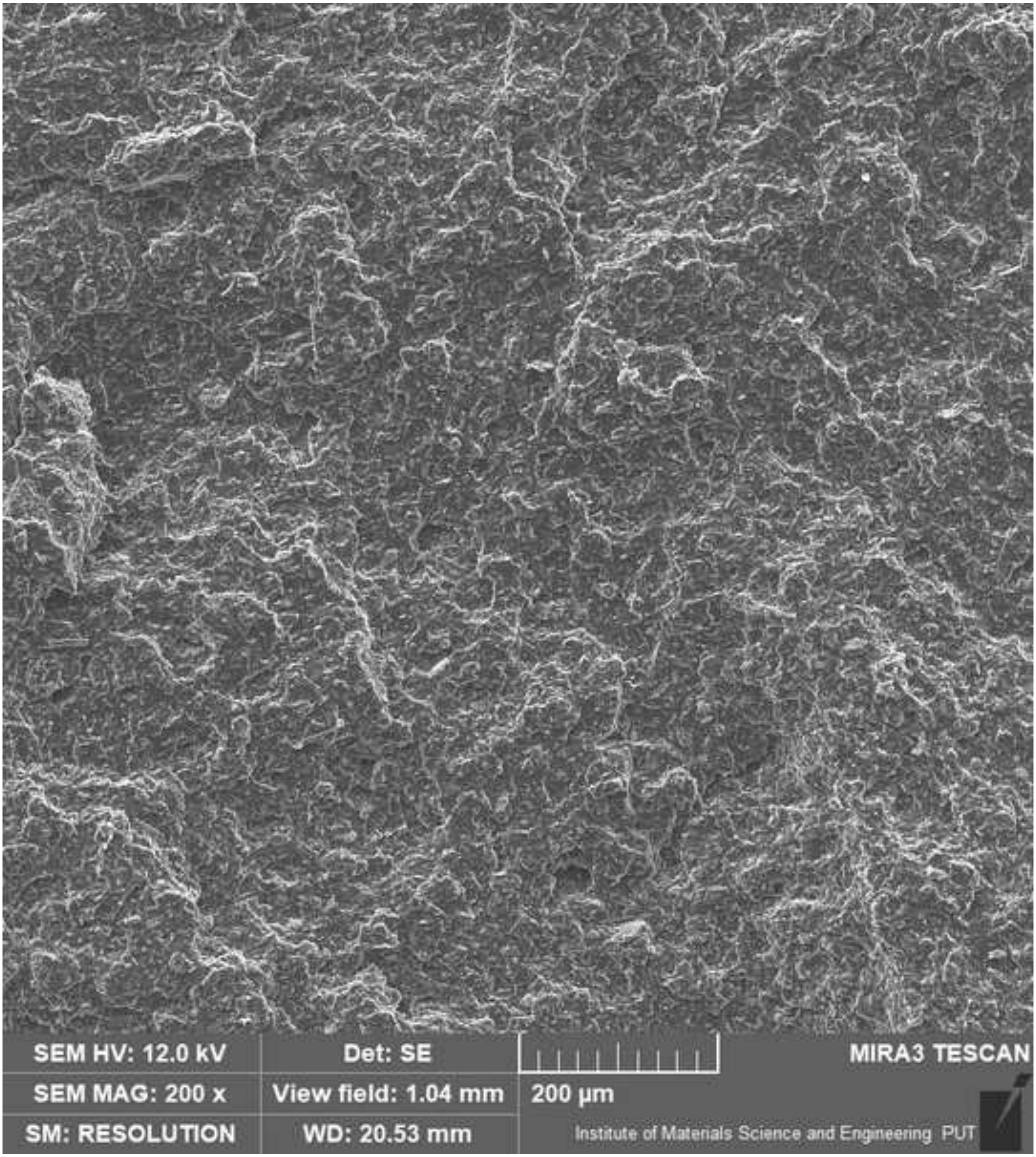
Click here to access/download;Figure;DP12540C7200.png

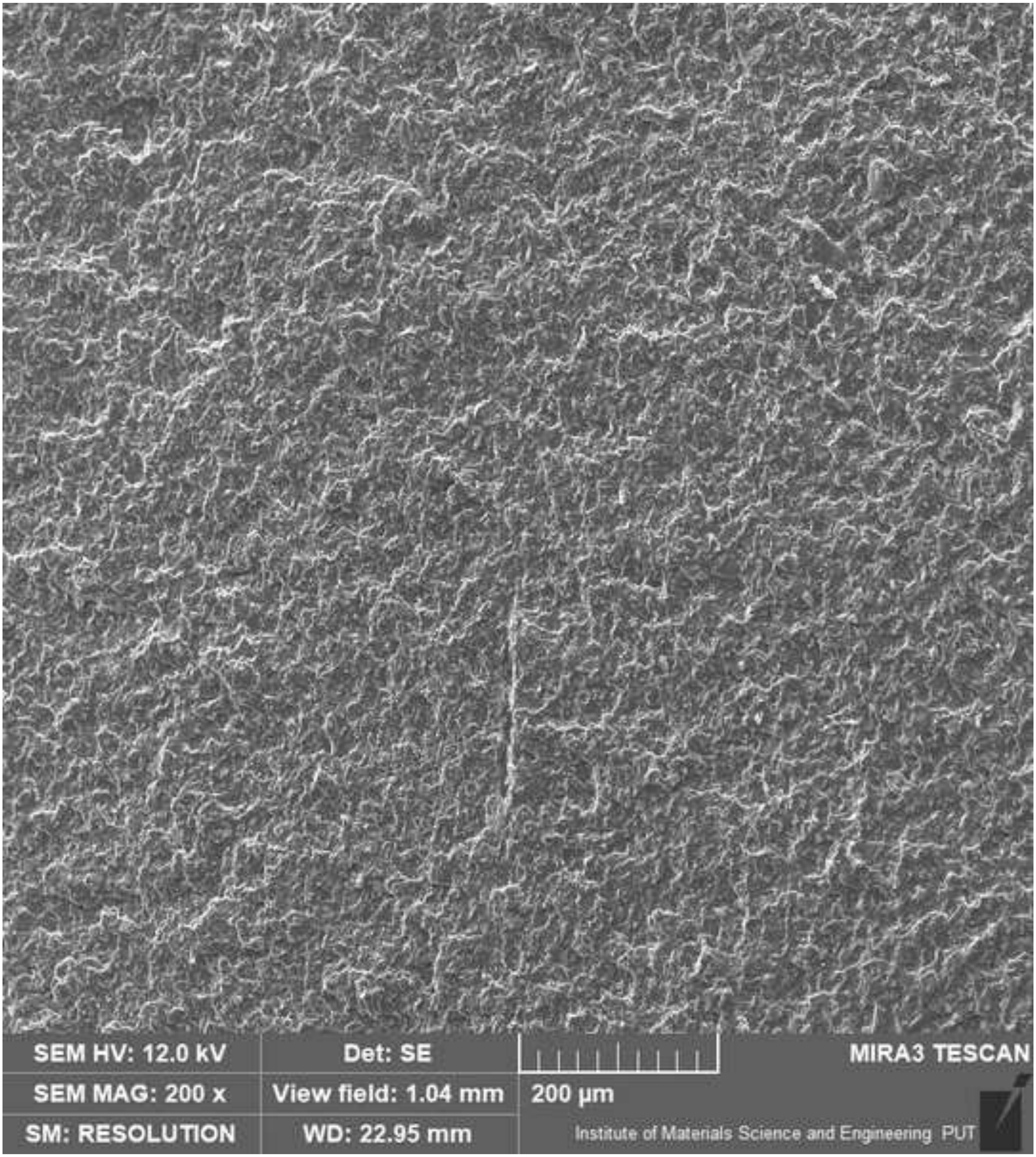


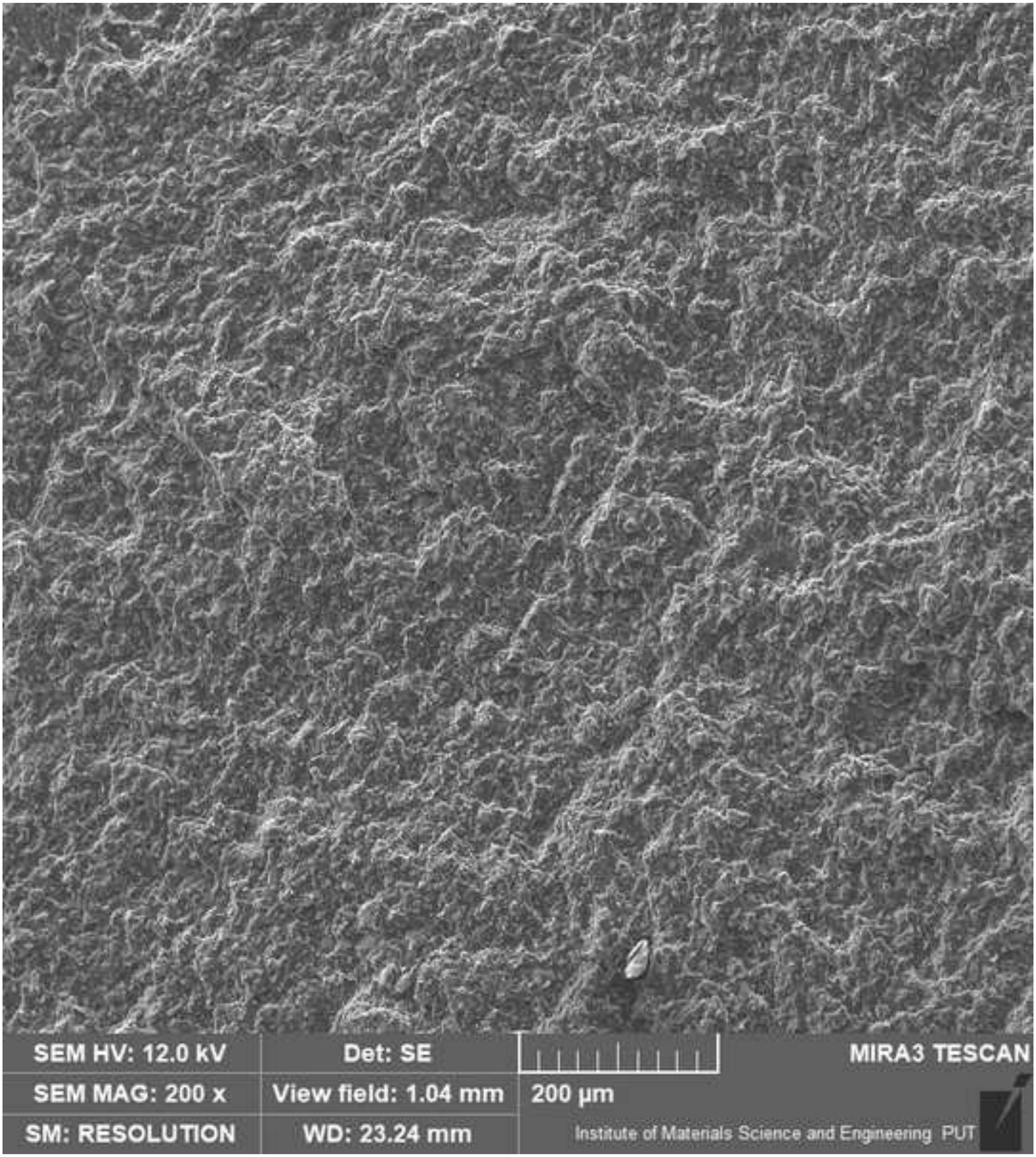
Click here to access/download;Figure;DP12560C14200.png

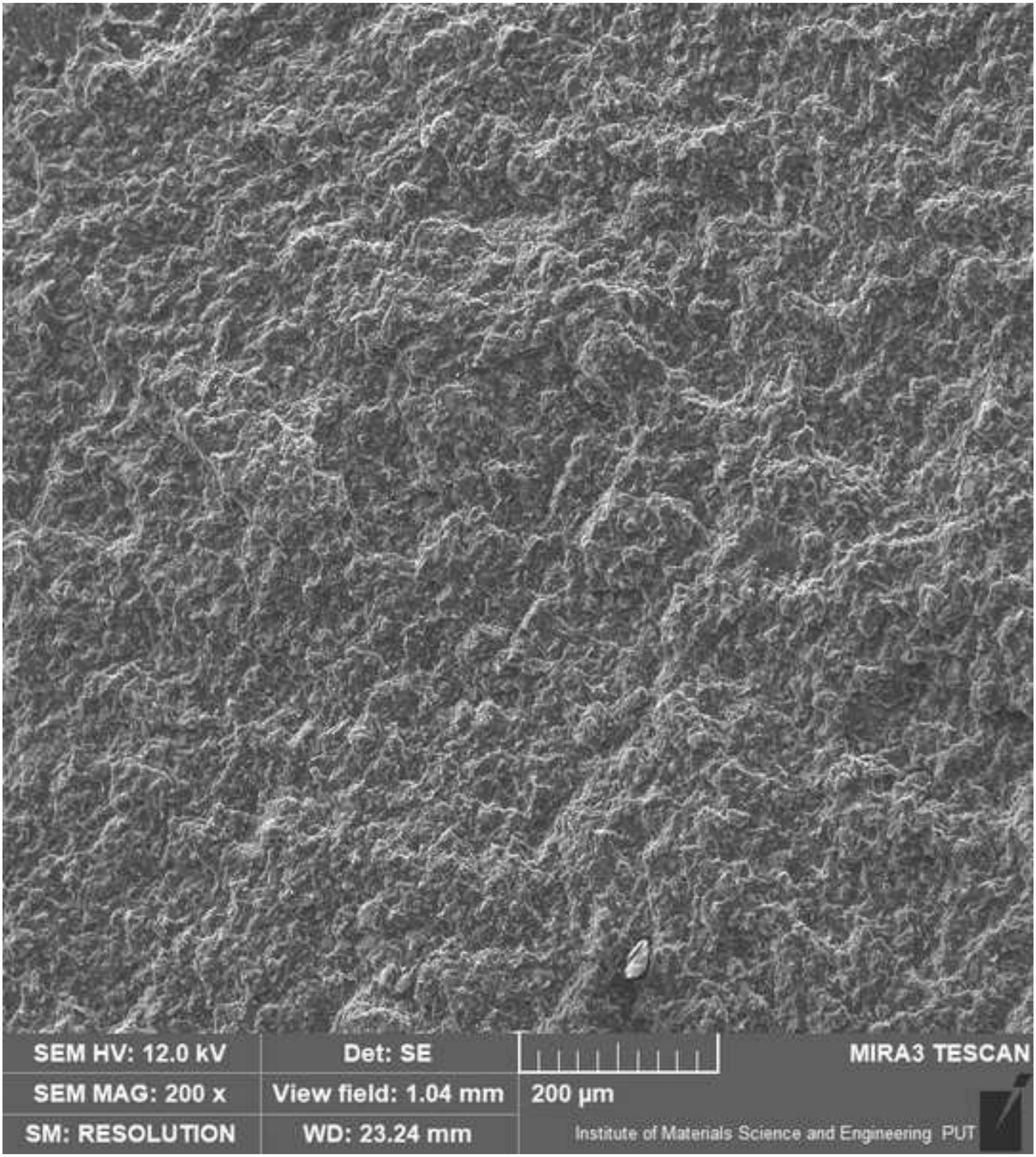



SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 22.45 mm
Institute of Materials Science and Engineering PUT

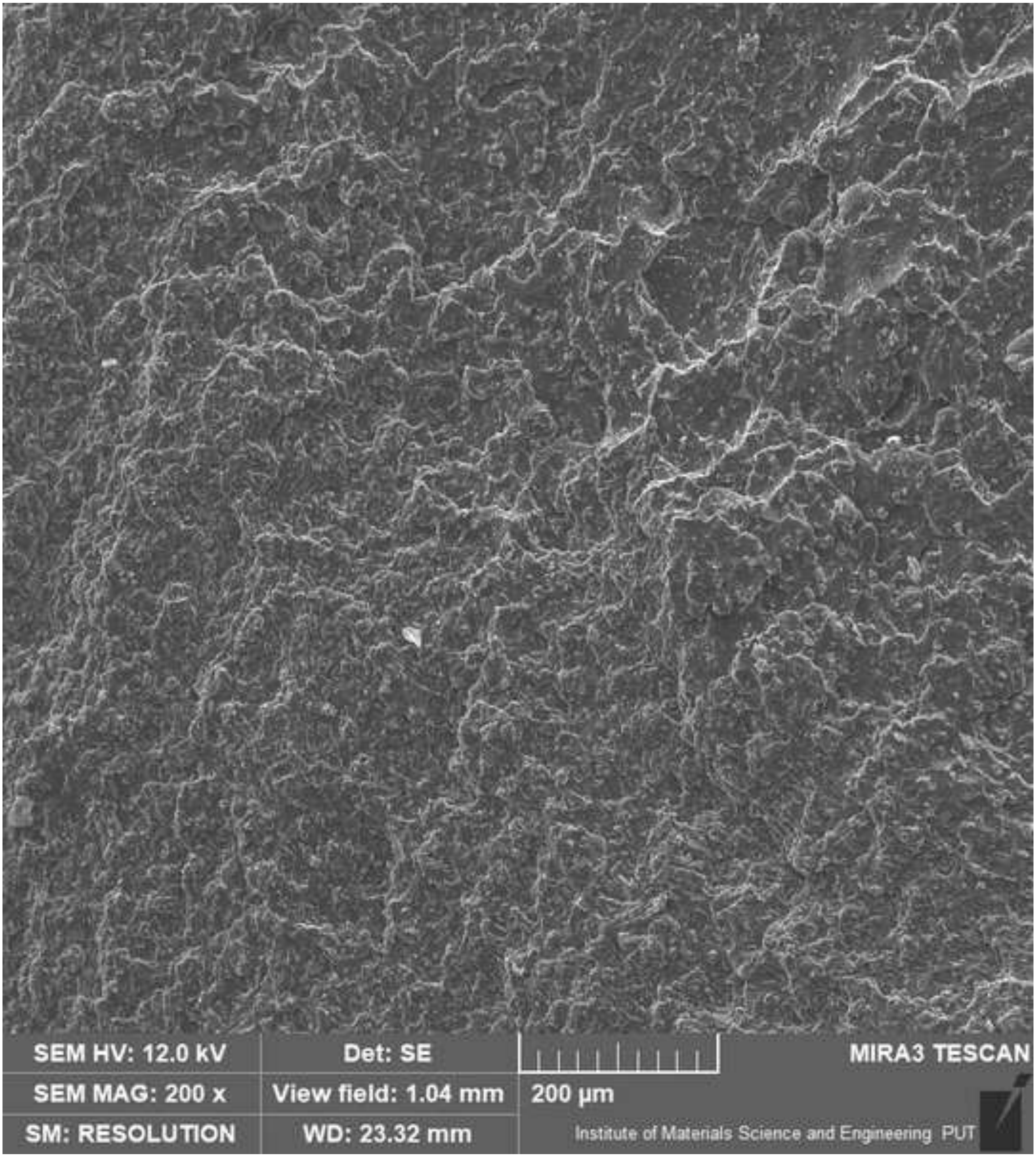

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 20.53 mm
Institute of Materials Science and Engineering PUT

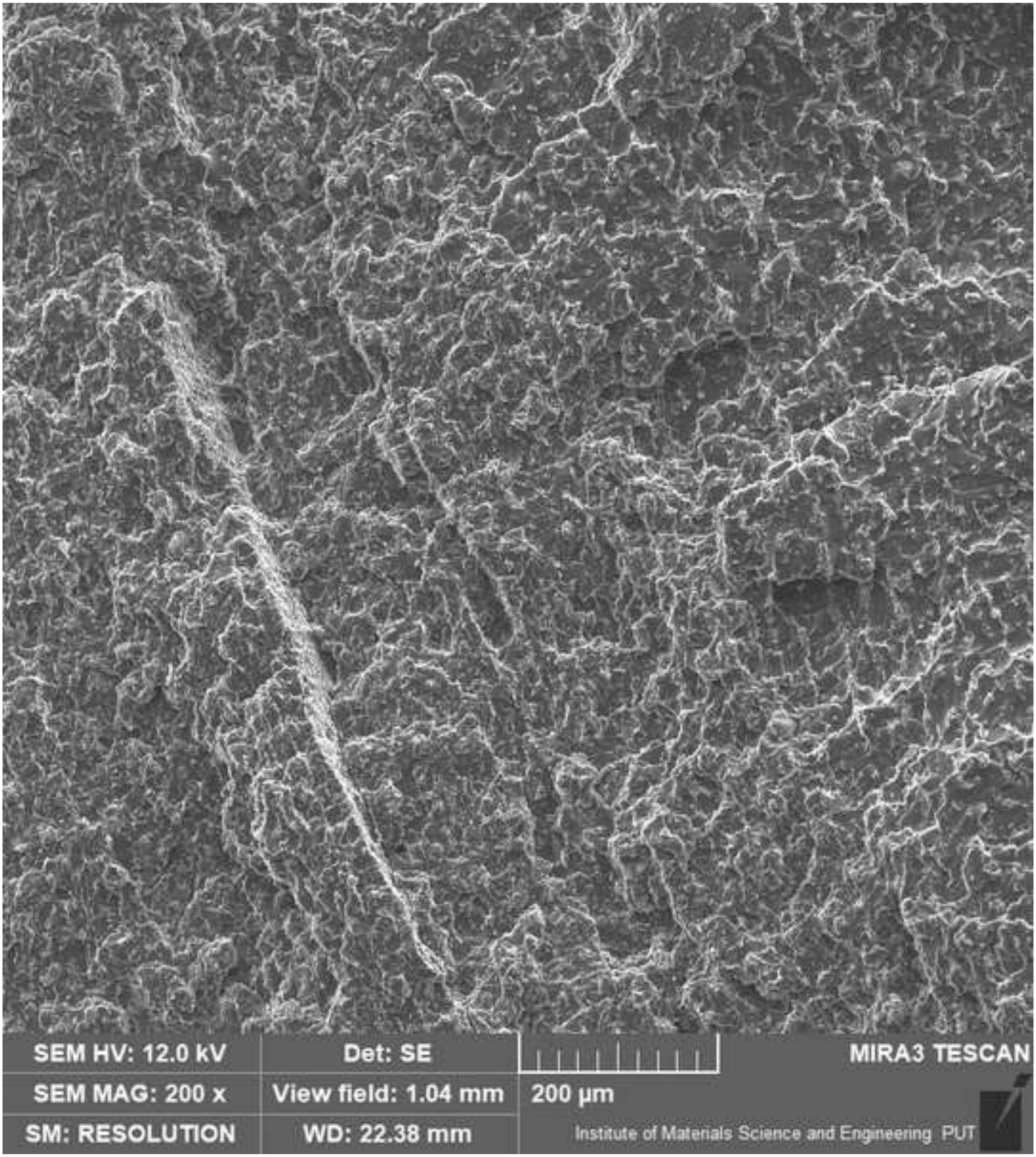

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 22.95 mm
Institute of Materials Science and Engineering PUT

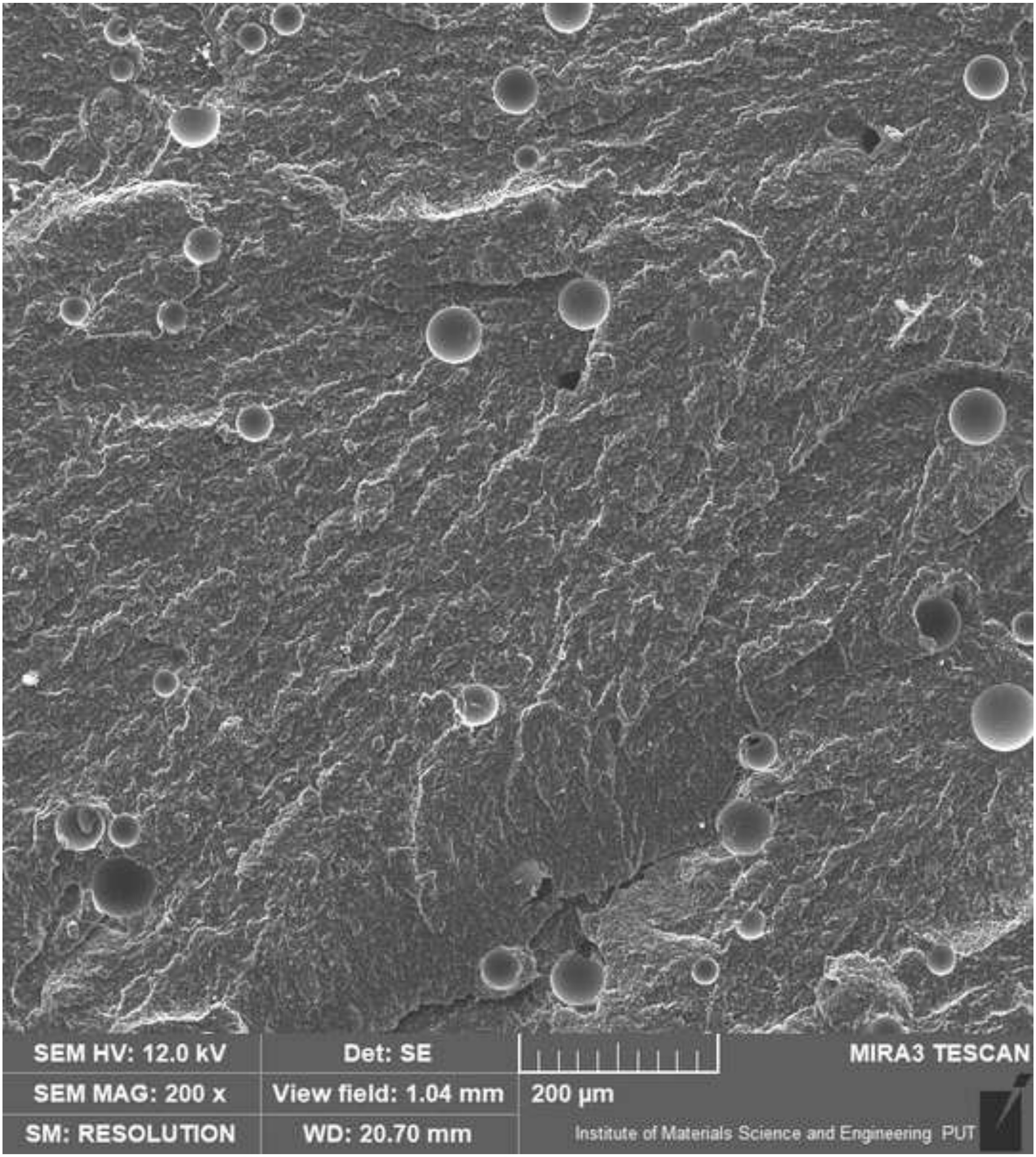

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 23.24 mm
Institute of Materials Science and Engineering PUT

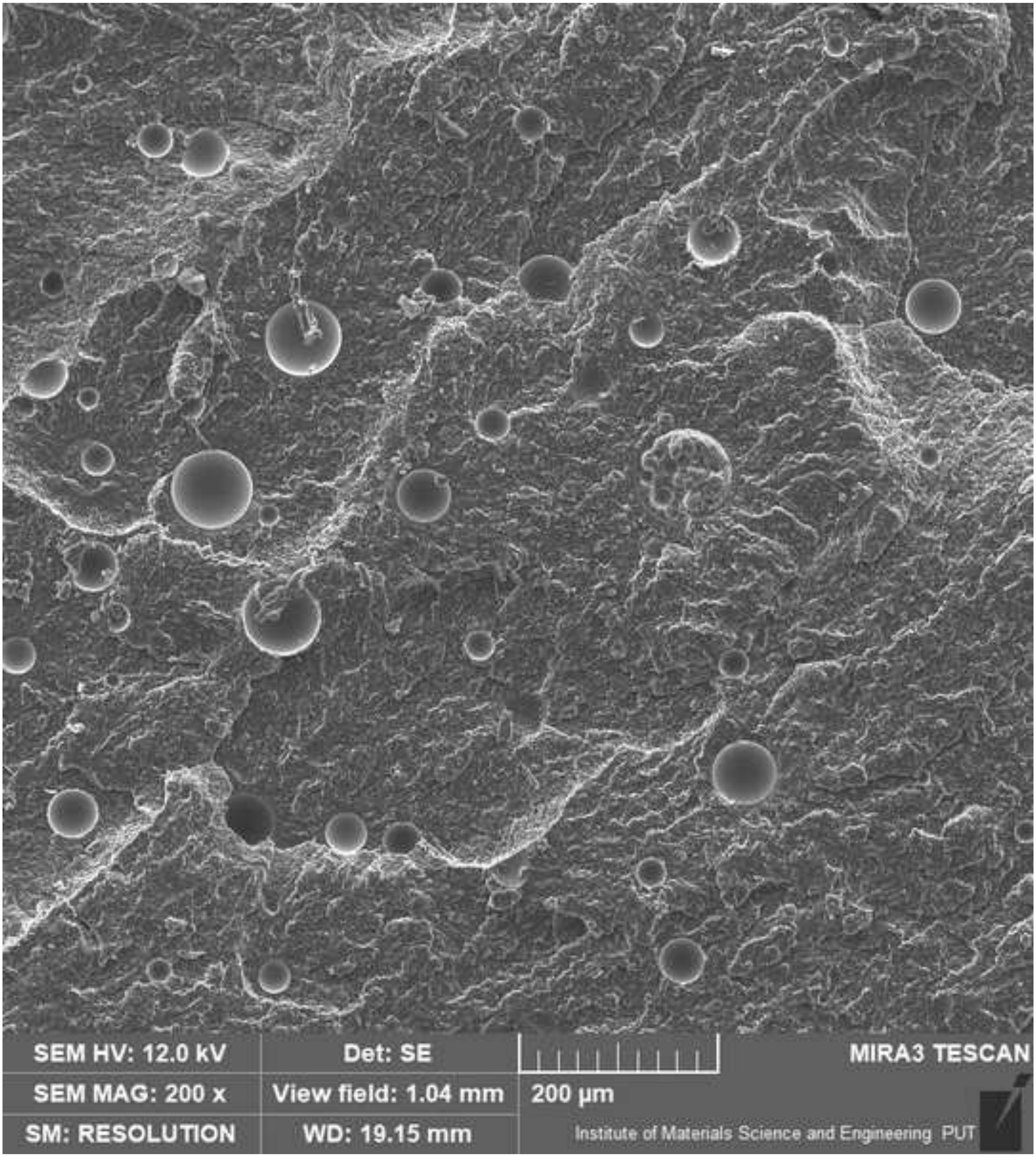

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 23.24 mm
Institute of Materials Science and Engineering PUT

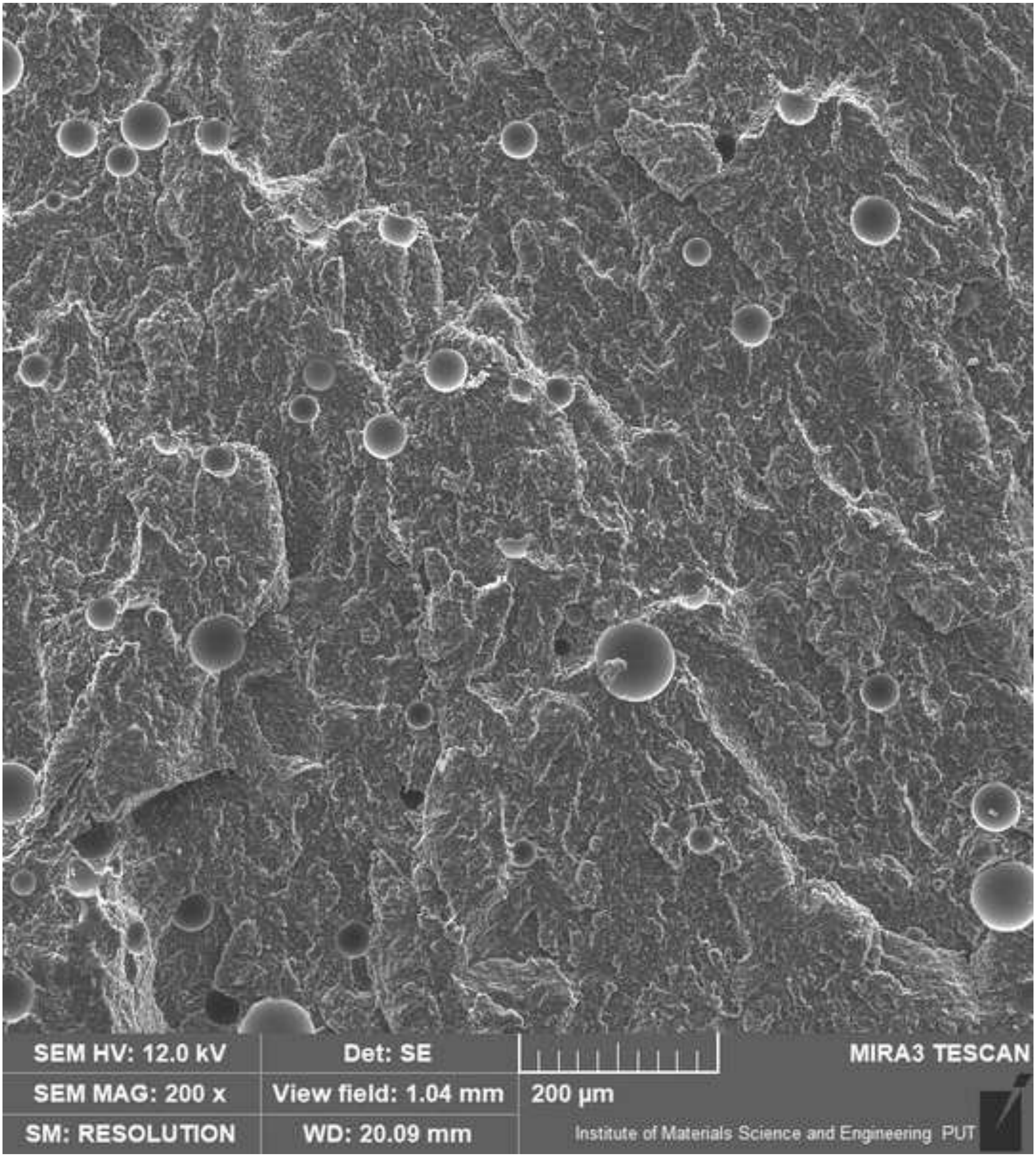
Click here to access/download;Figure;DP12580C7200.png

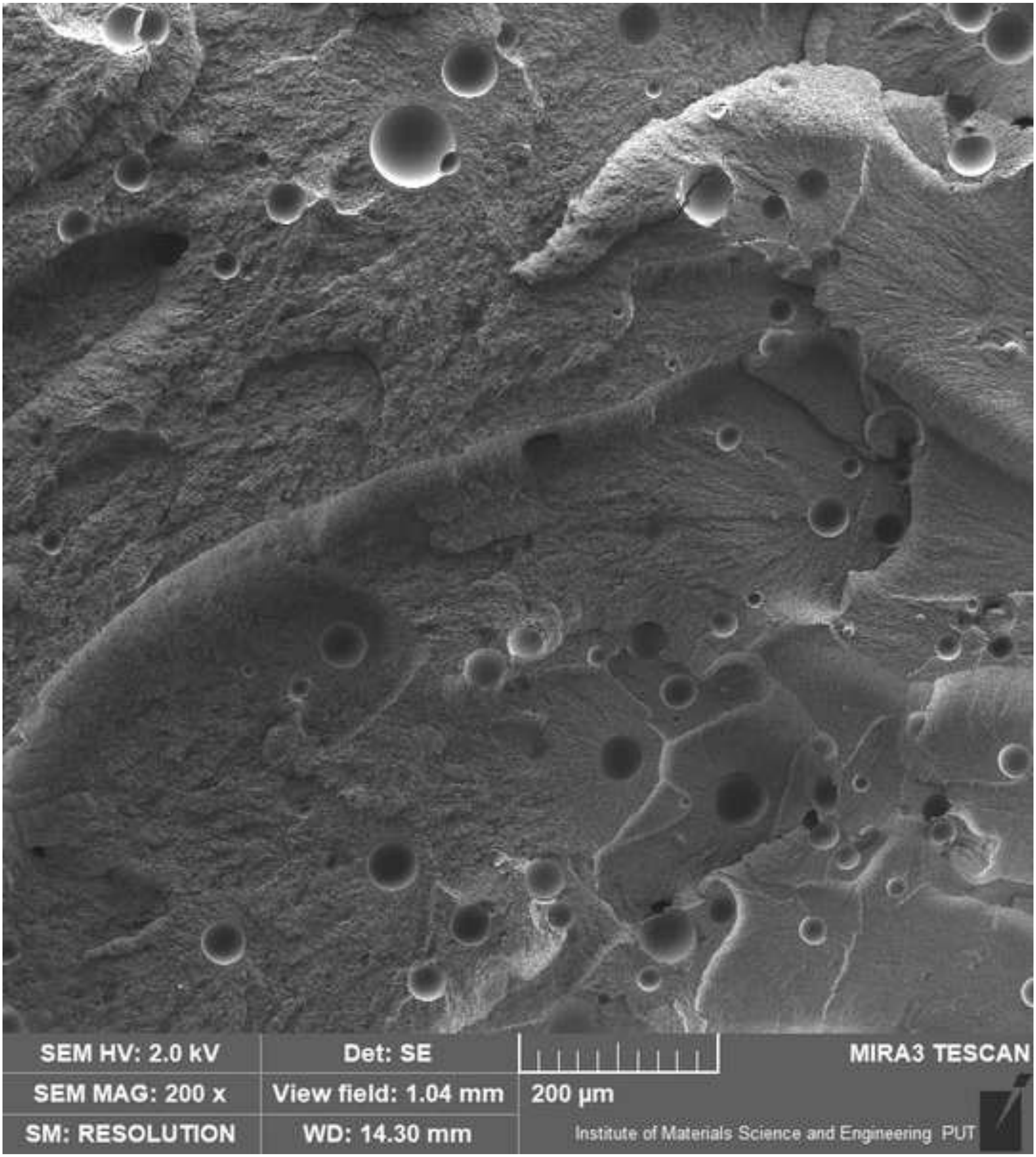


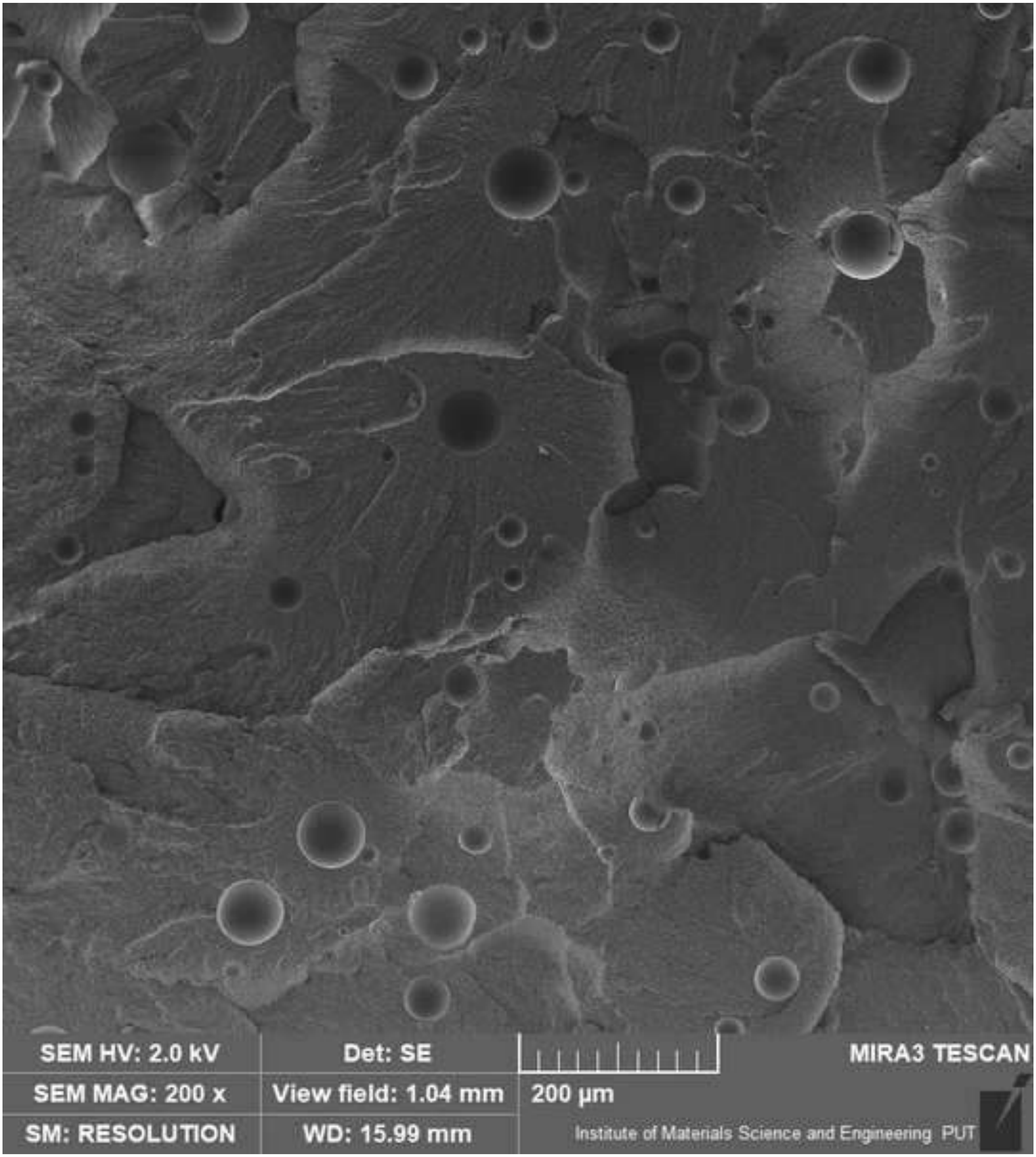

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 22.38 mm
Institute of Materials Science and Engineering PUT

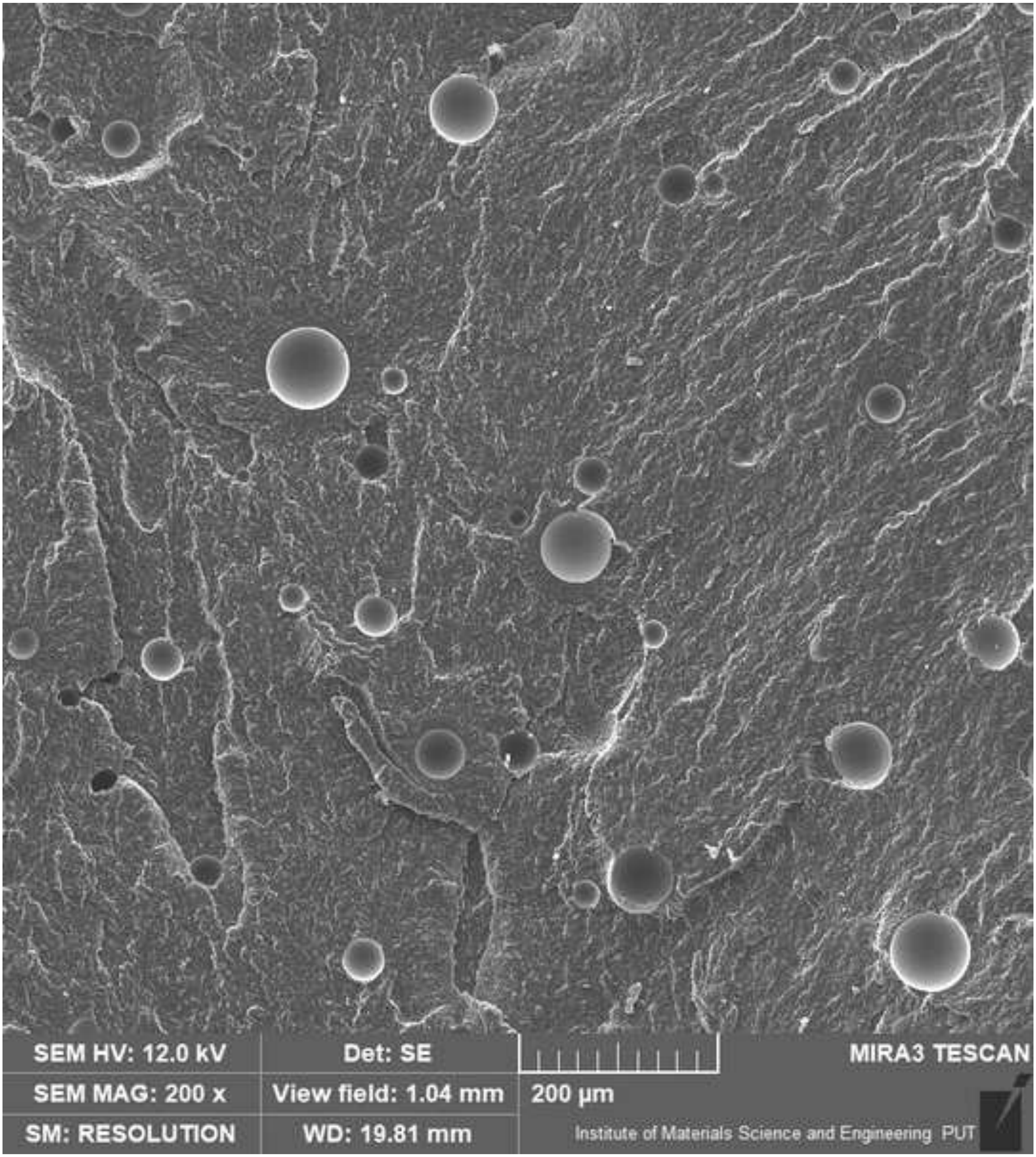

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 20.70 mm
Institute of Materials Science and Engineering PUT

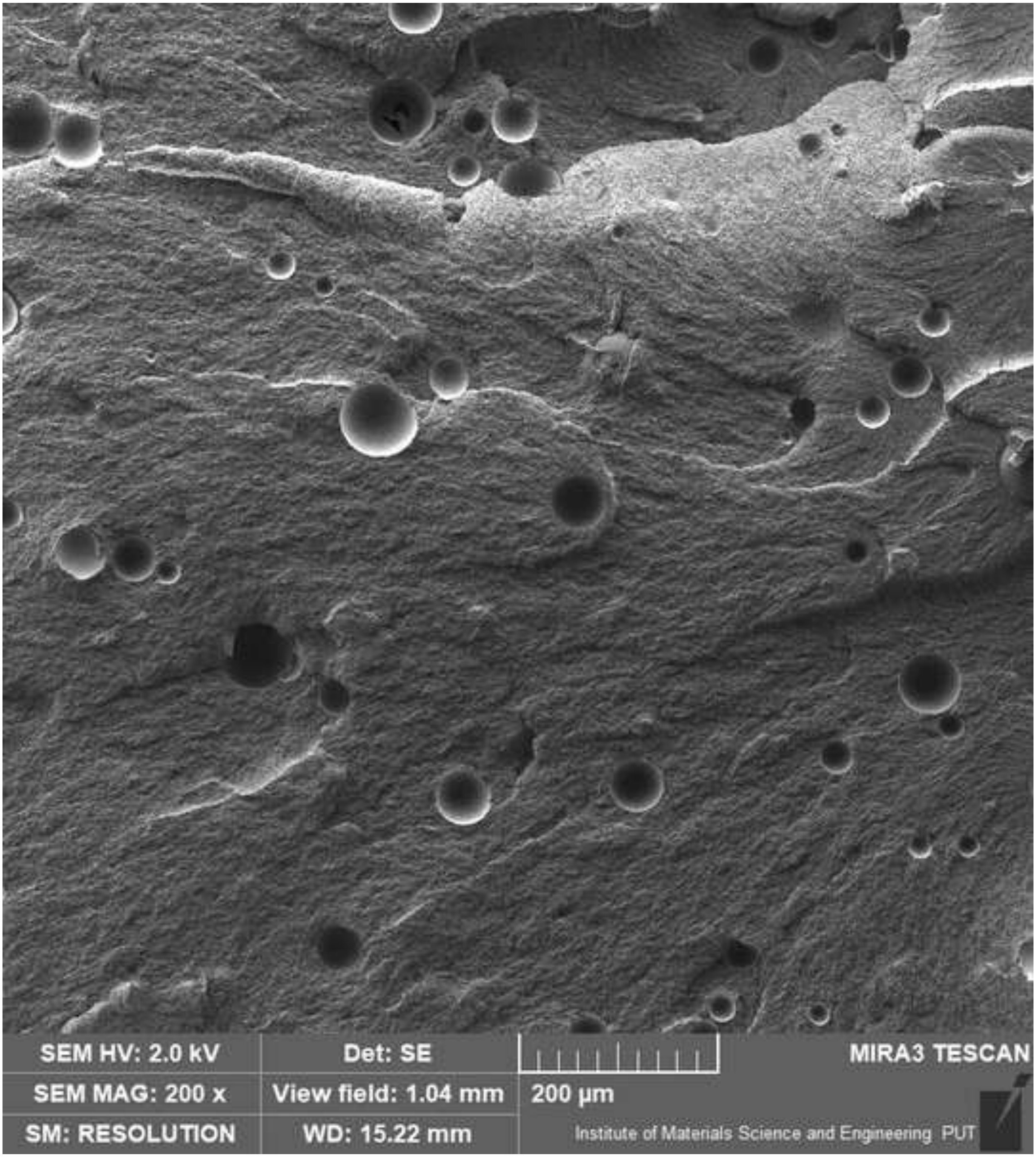

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 19.15 mm
Institute of Materials Science and Engineering PUT

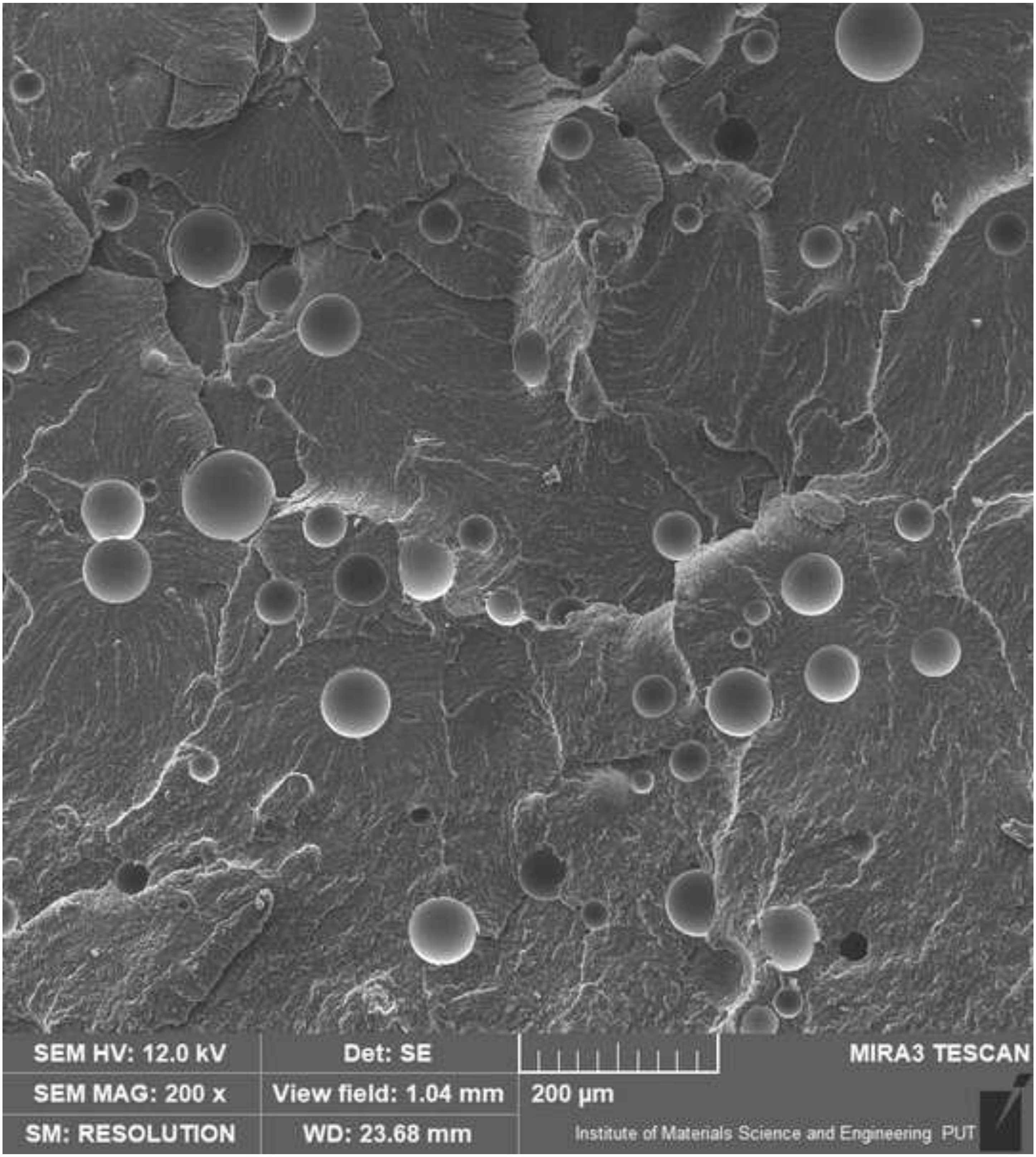

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 20.09 mm
Institute of Materials Science and Engineering PUT

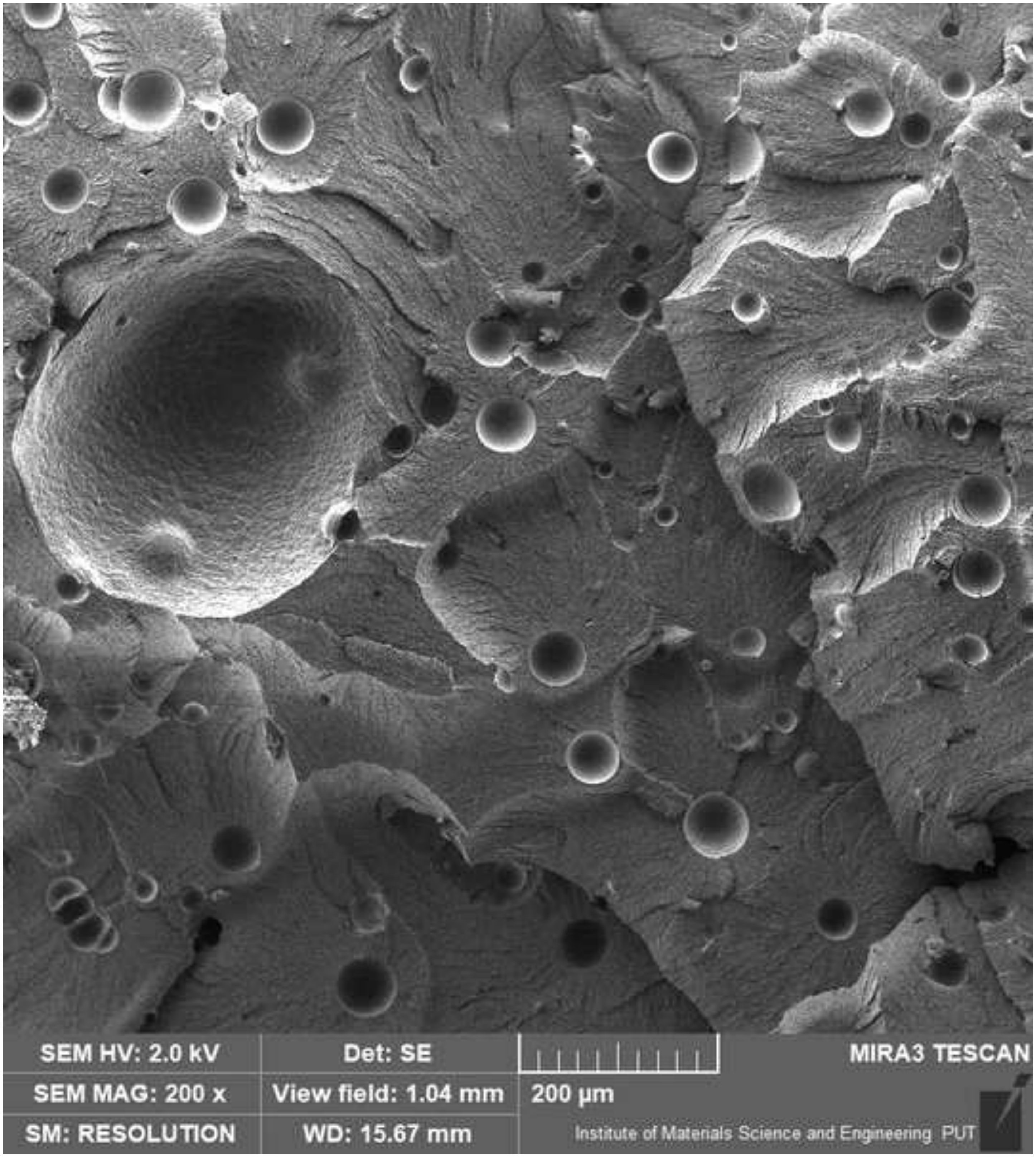

SEM HV: 2.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 14.30 mm
Institute of Materials Science and Engineering PUT


SEM HV: 2.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 15.99 mm
Institute of Materials Science and Engineering PUT

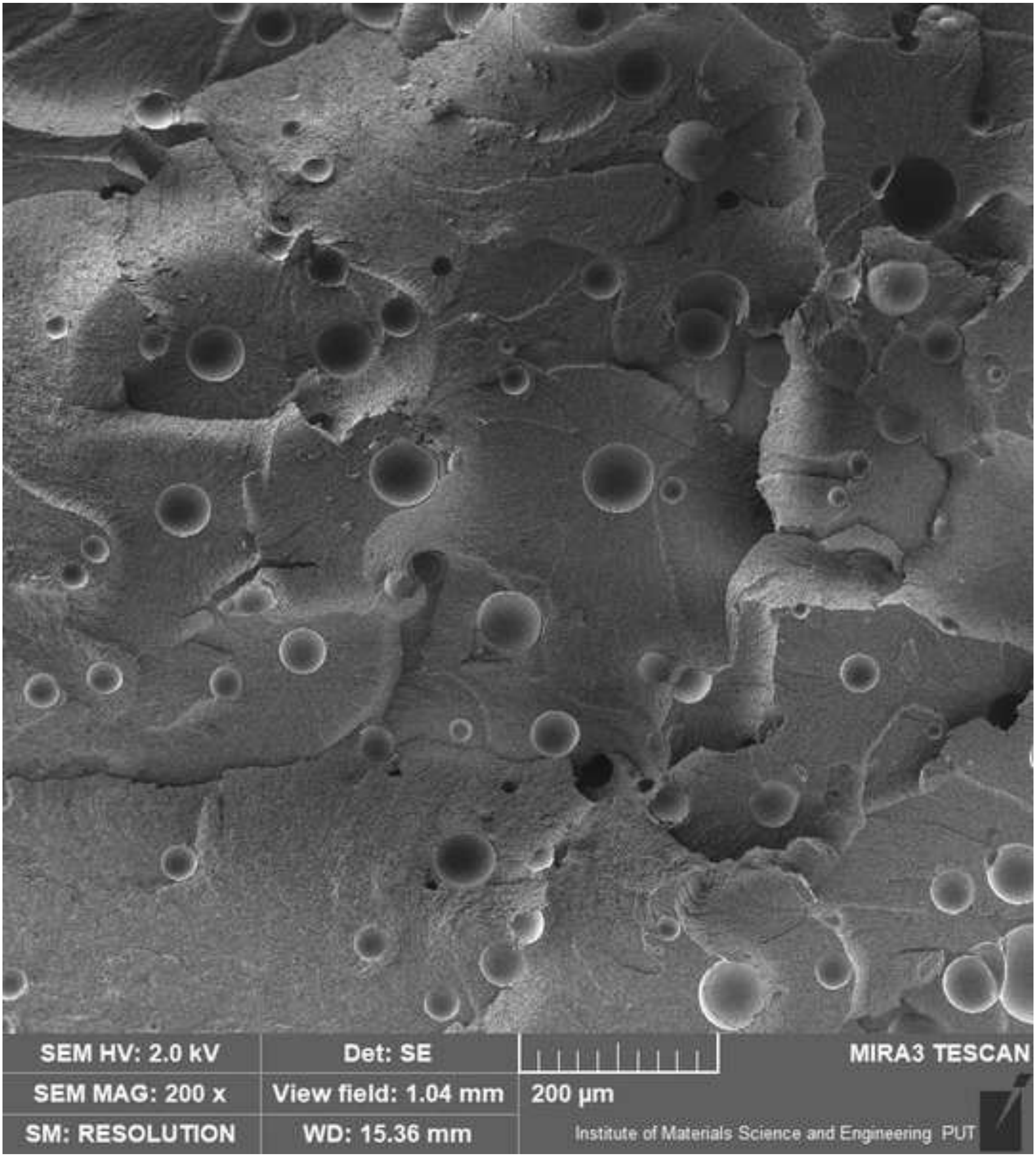

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 19.81 mm
Institute of Materials Science and Engineering PUT

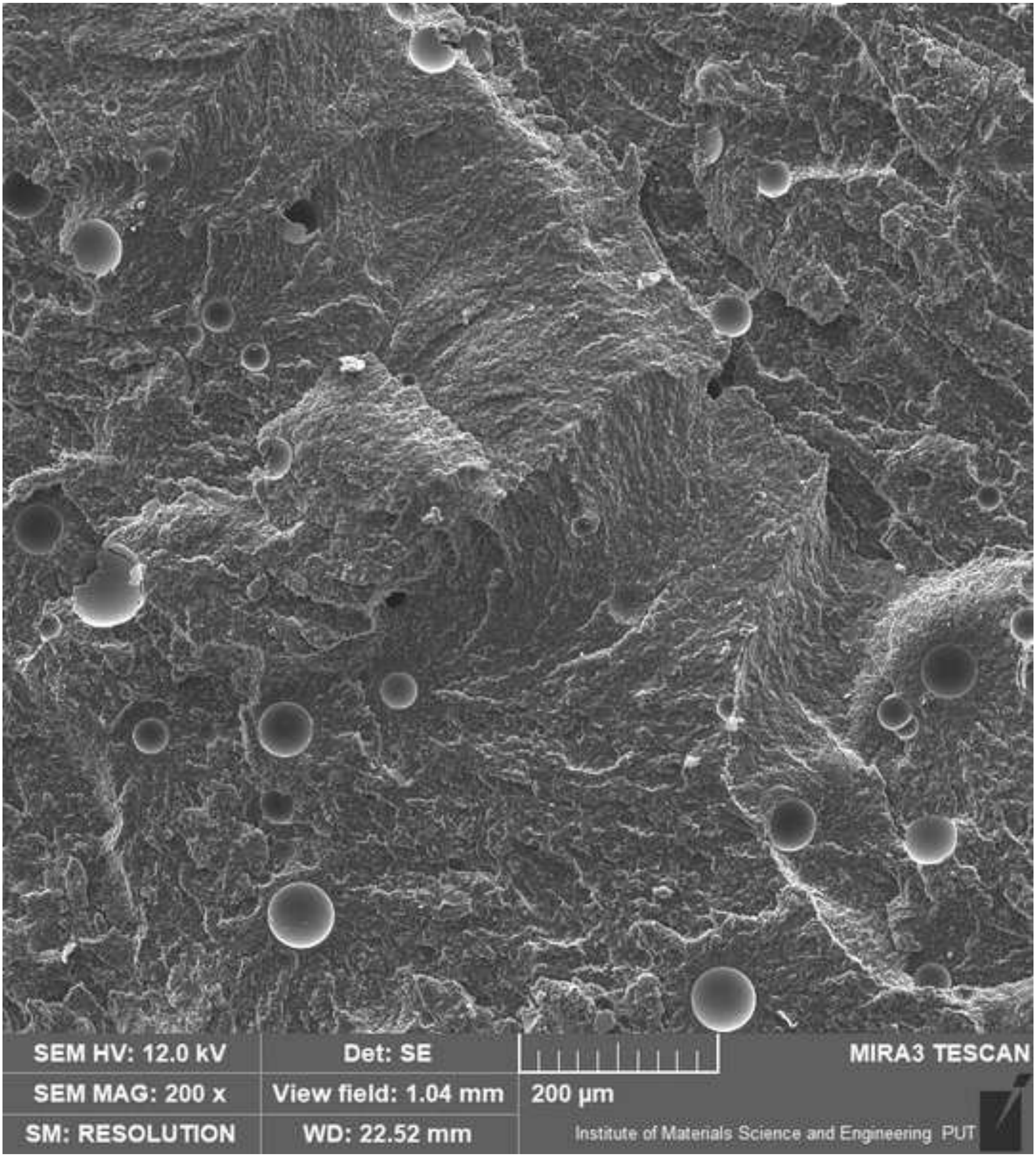

SEM HV: 2.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 15.22 mm
Institute of Materials Science and Engineering PUT

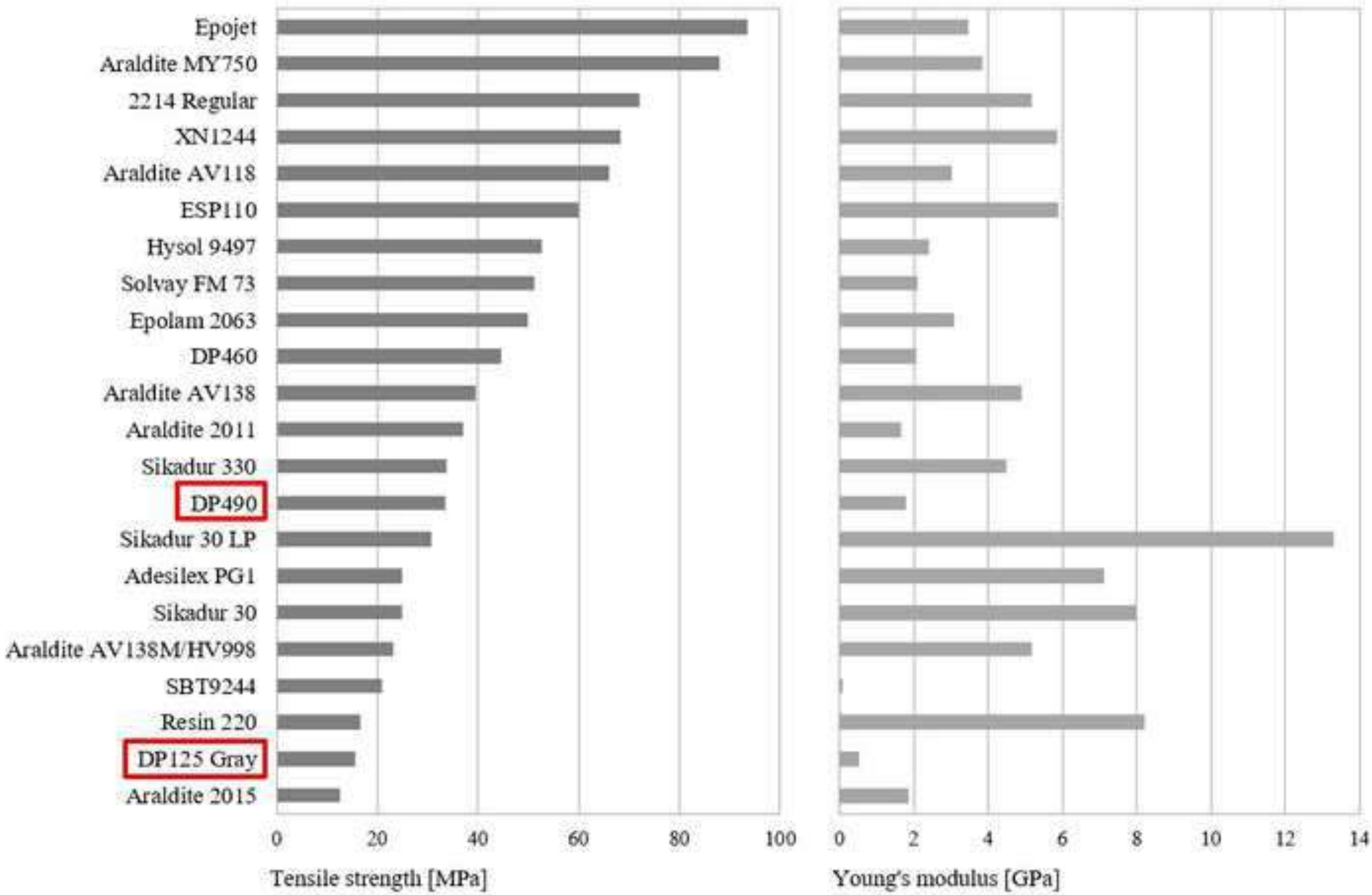

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 23.68 mm
Institute of Materials Science and Engineering PUT

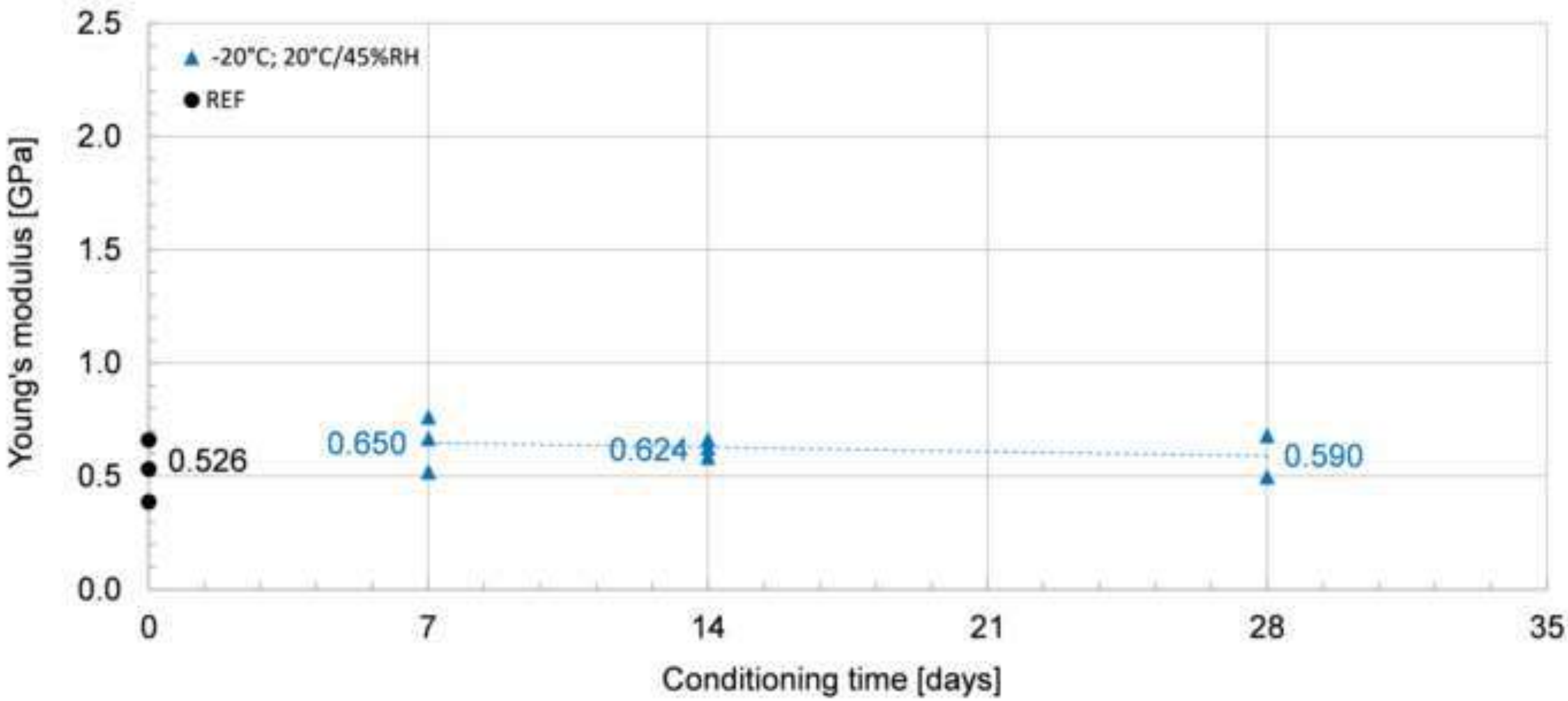

SEM HV: 2.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 μm
SM: RESOLUTION
WD: 15.67 mm
Institute of Materials Science and Engineering PUT

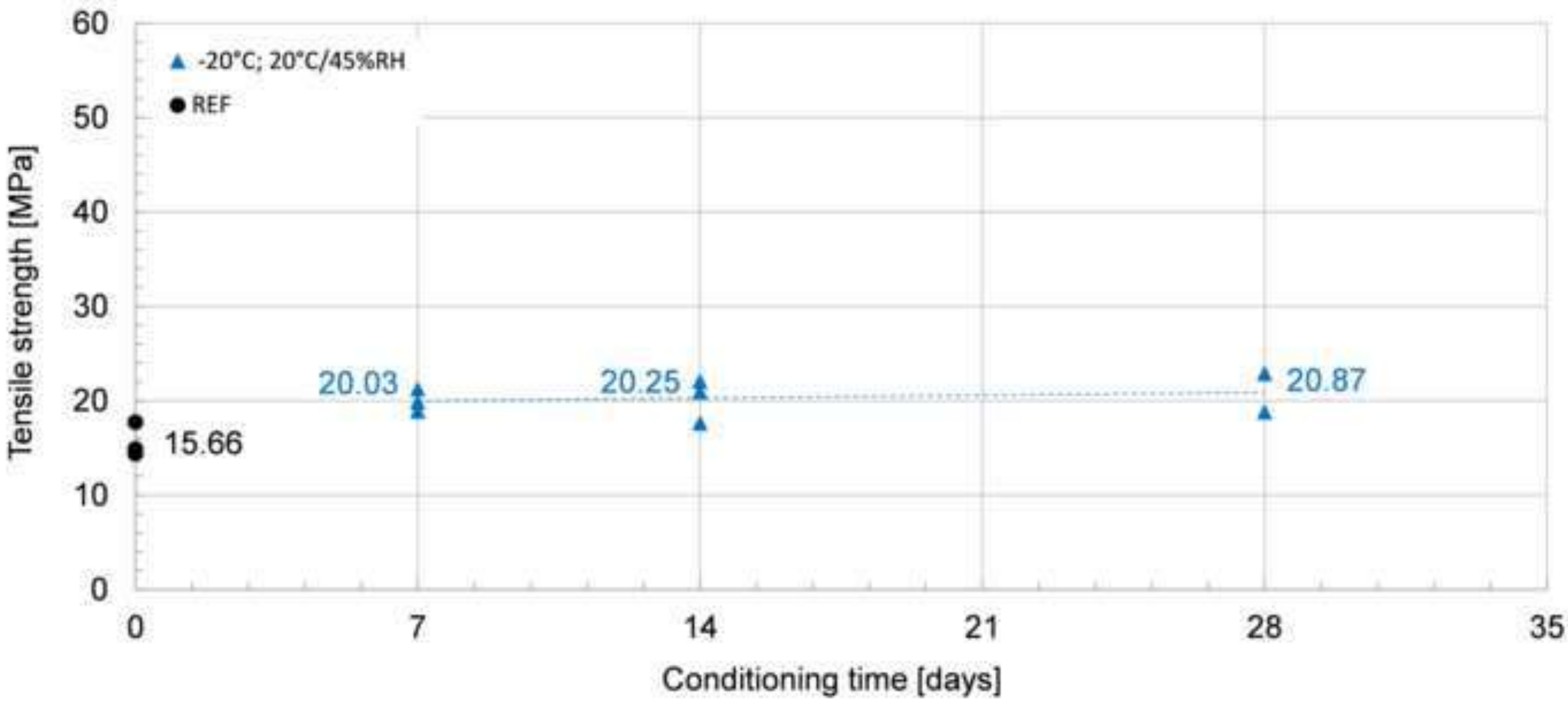
Click here to access/download;Figure;DP49080C7200.png



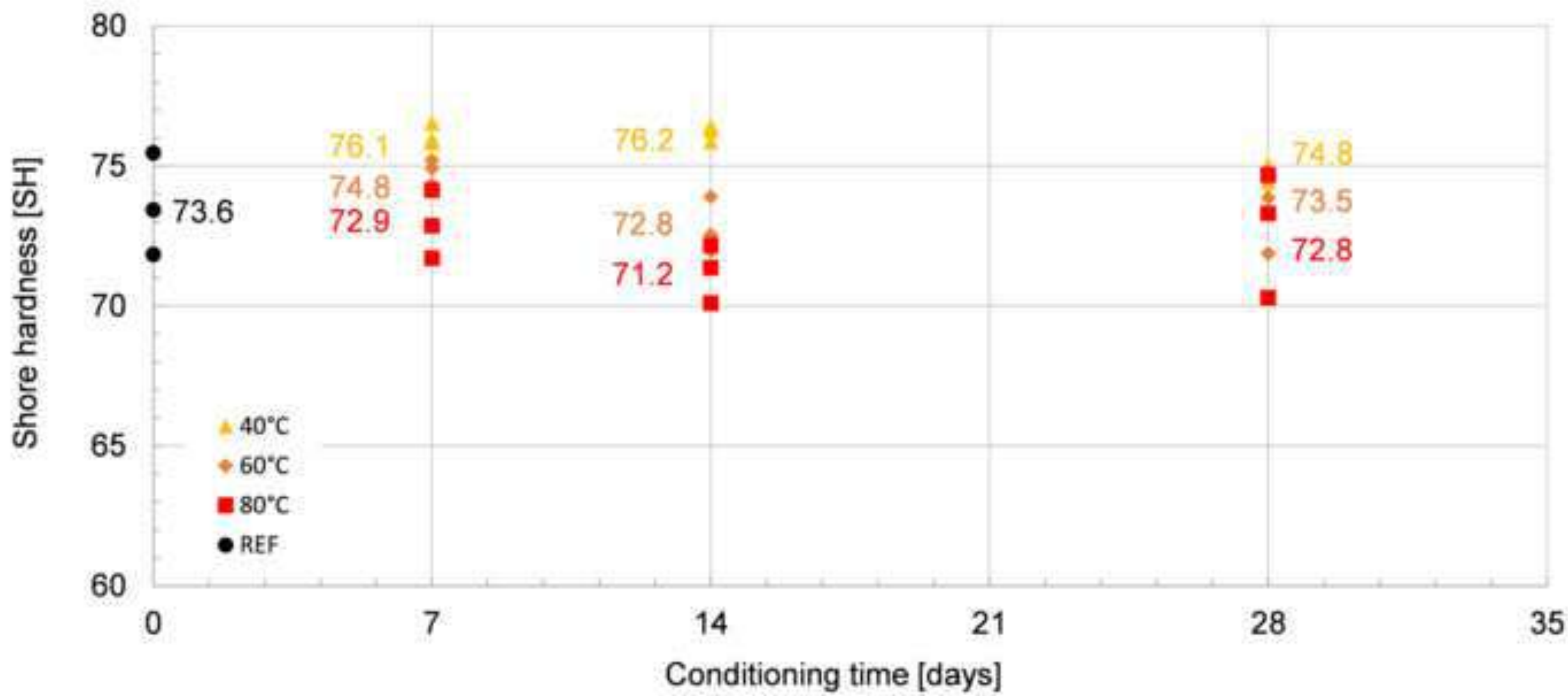

SEM HV: 12.0 kV
Det: SE
MIRA3 TESCAN
SEM MAG: 200 x
View field: 1.04 mm
200 µm
SM: RESOLUTION
WD: 22.52 mm
Institute of Materials Science and Engineering PUT


Epojet
Araldite MY750
2214 Regular
XN1244
Araldite AV118
ESP110
Hysol 9497
Solvay FM 73
Epolam 2063
DP460
Araldite AV138
Araldite 2011
Sikadur 330
DP490
Sikadur 30 LP
Adesilex PG1
Sikadur 30
Araldite AV138M/HV998
SBT9244
Resin 220
DP125 Gray
Araldite 2015
0 20 40 60 80 100
Tensile strength [MPa]
0 2 4 6 8 10 12 14
Young's modulus [GPa]


-20°C; 20°C/45%RH
REF
Young's modulus [GPa]
Conditioning time [days]
0.526
0.650
0.624
0.590
2.5
2.0
1.5
1.0
0.5
0.0
0
7
14
21
28
35

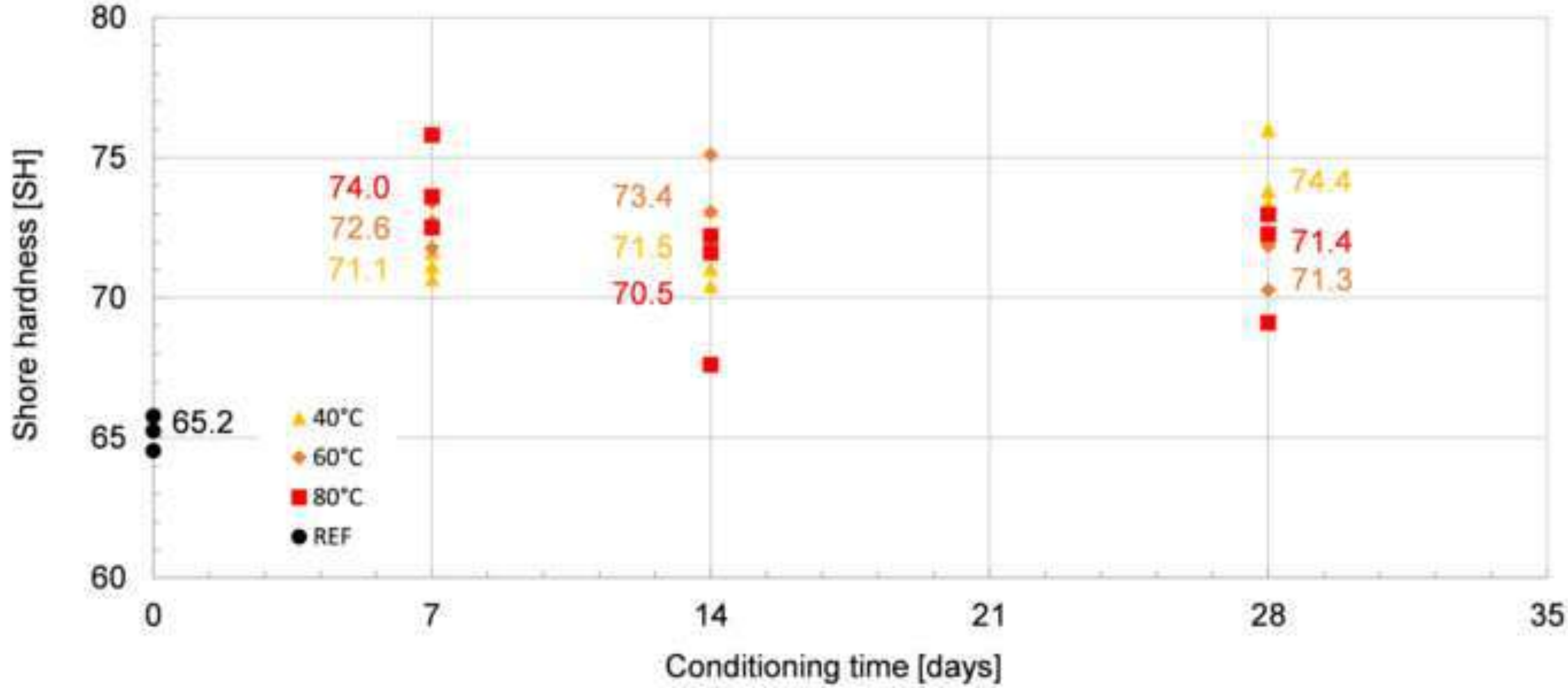


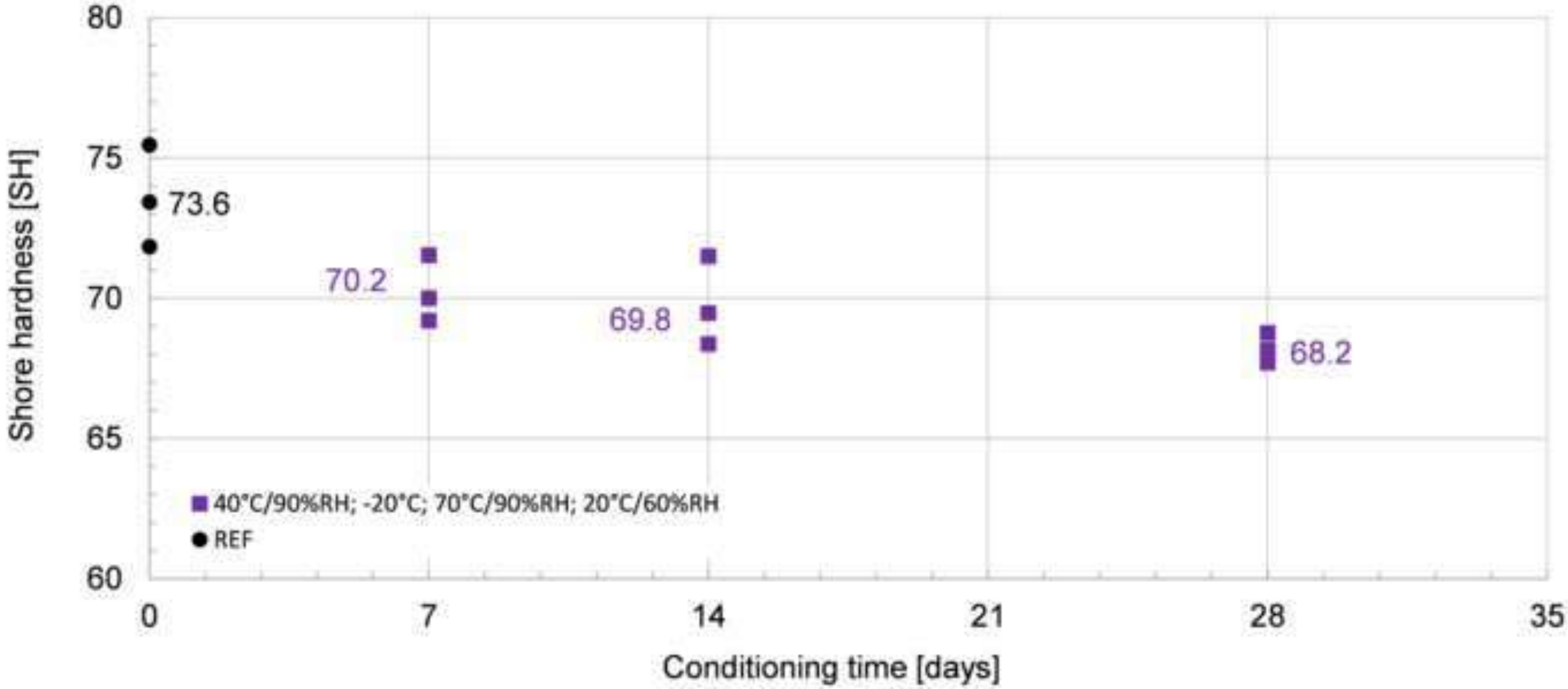
-20°C; 20°C/45%RH
REF
Tensile strength [MPa]
60
50
40
30
20
10
0
20.03
20.25
20.87
15.66
0
7
14
21
28
35
Conditioning time [days]



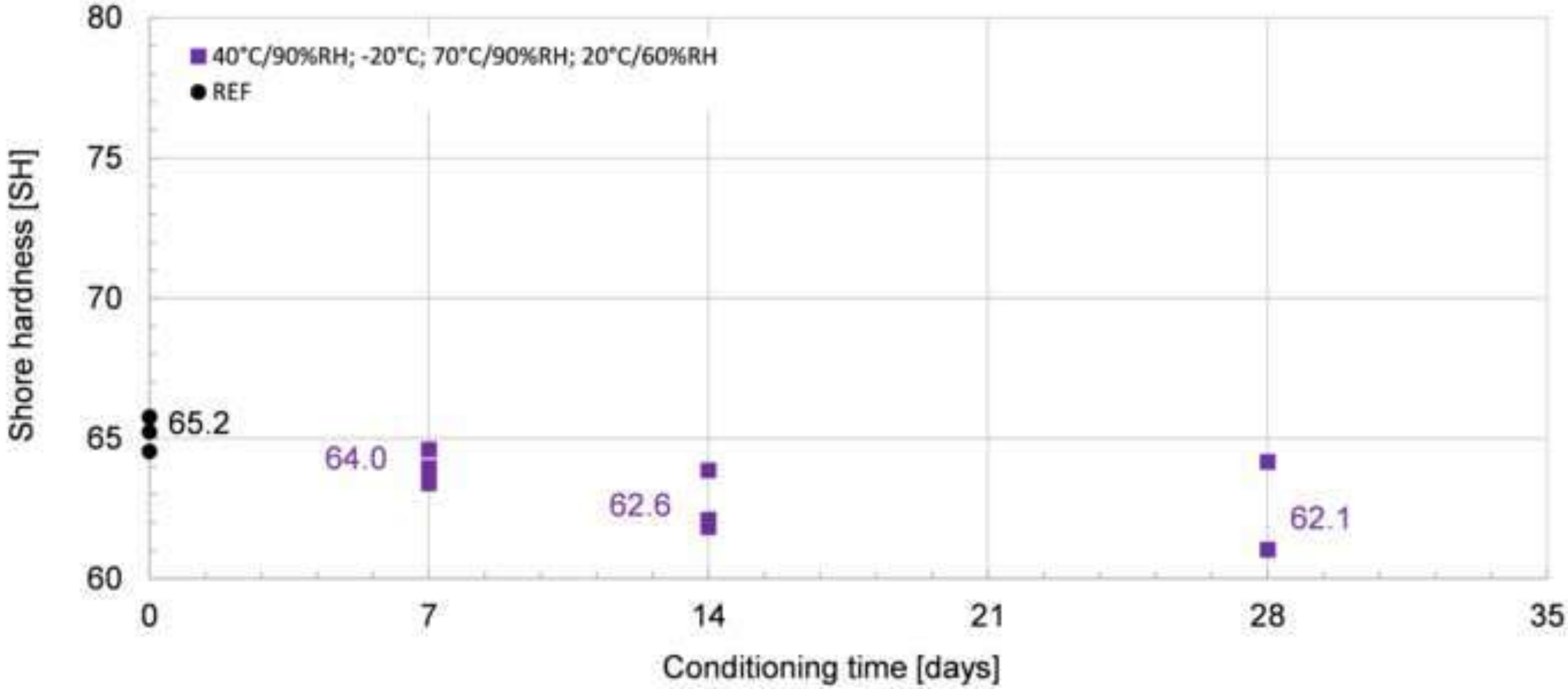
Shore hardness [SH]
80
75
70
65
60
0
7
14
21
28
35
Conditioning time [days]
73.6
76.1
74.8
72.9
76.2
72.8
71.2
74.8
73.5
72.8
40°C
60°C
80°C
REF

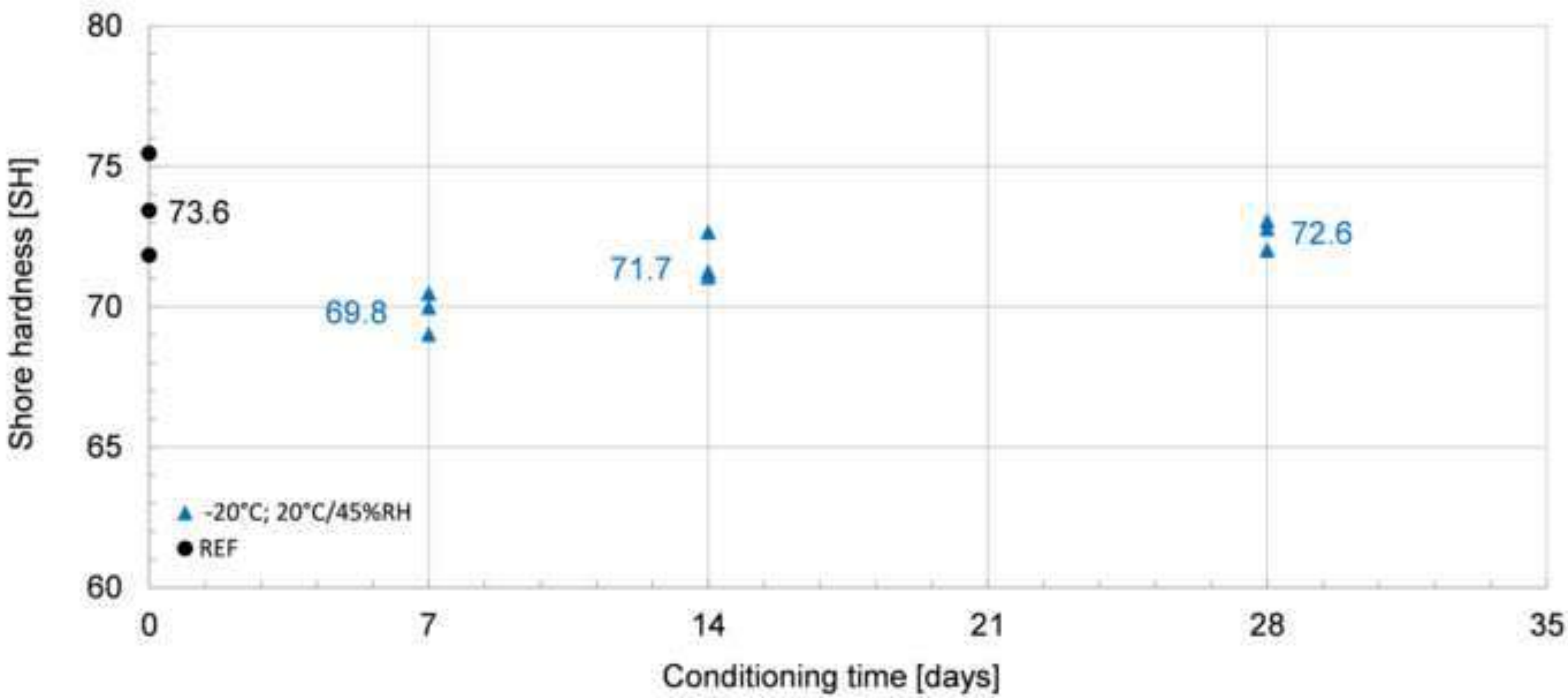
Click here to access/download;Figure;fig11b.png



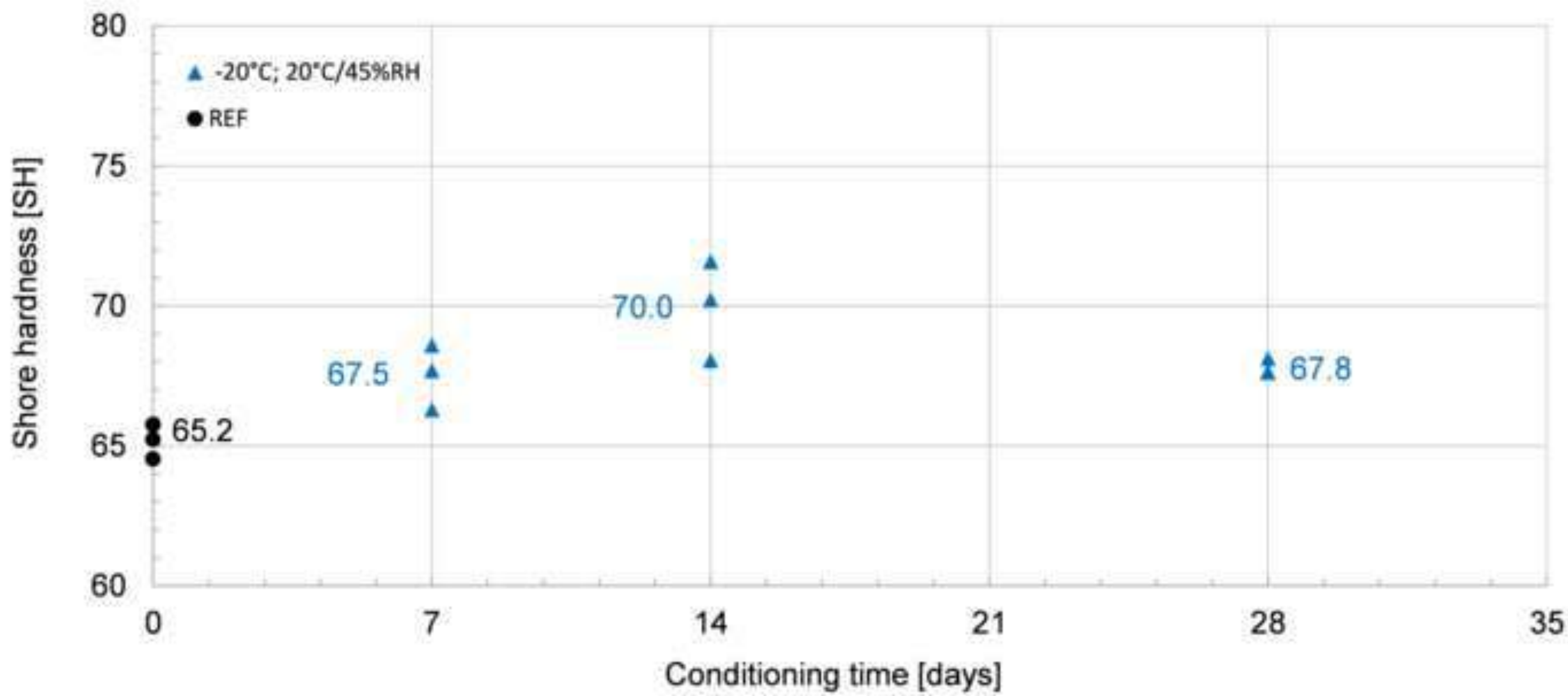

80
75
70
65
60
Shore hardness [SH]
73.6
70.2
69.8
68.2
40°C/90%RH; -20°C; 70°C/90%RH; 20°C/60%RH
REF
0
7
14
21
28
35
Conditioning time [days]

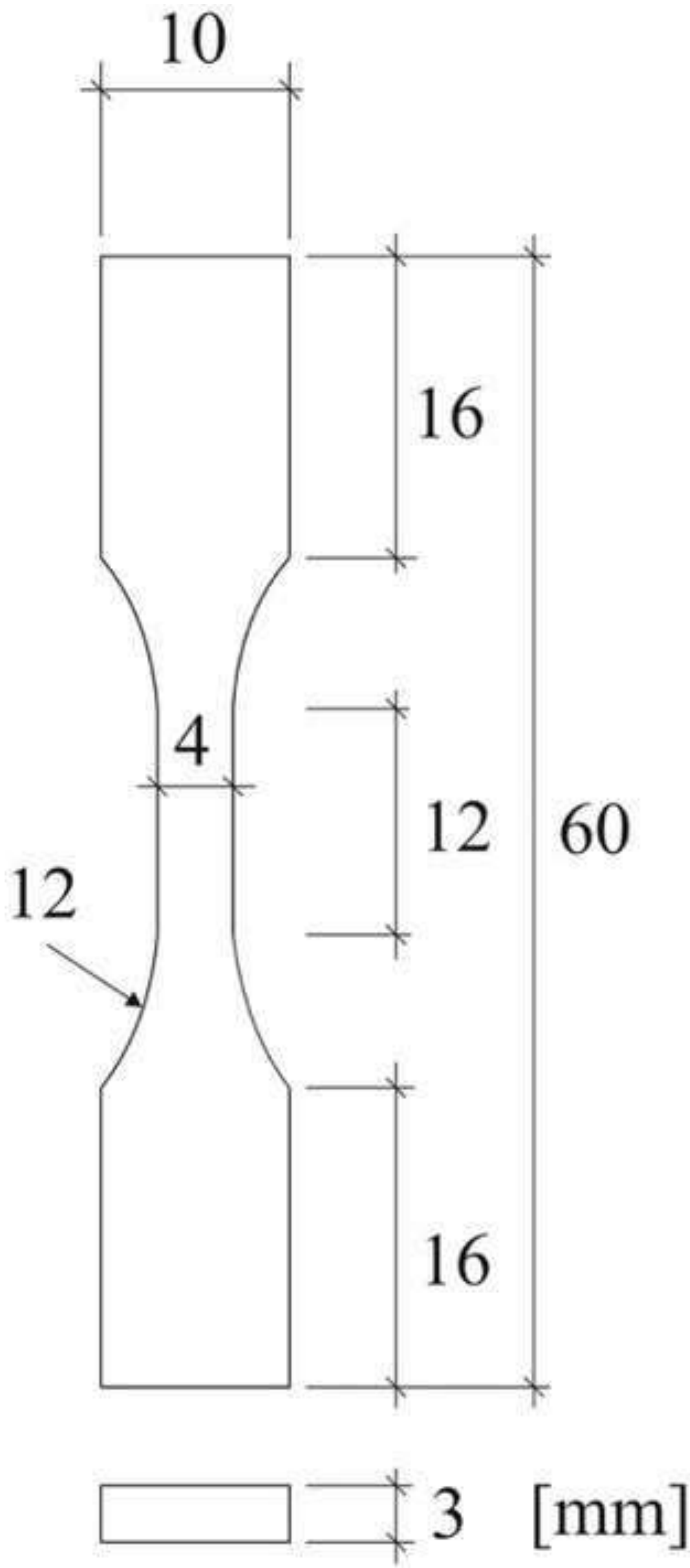
Click here to access/download;Figure;fig12b.png



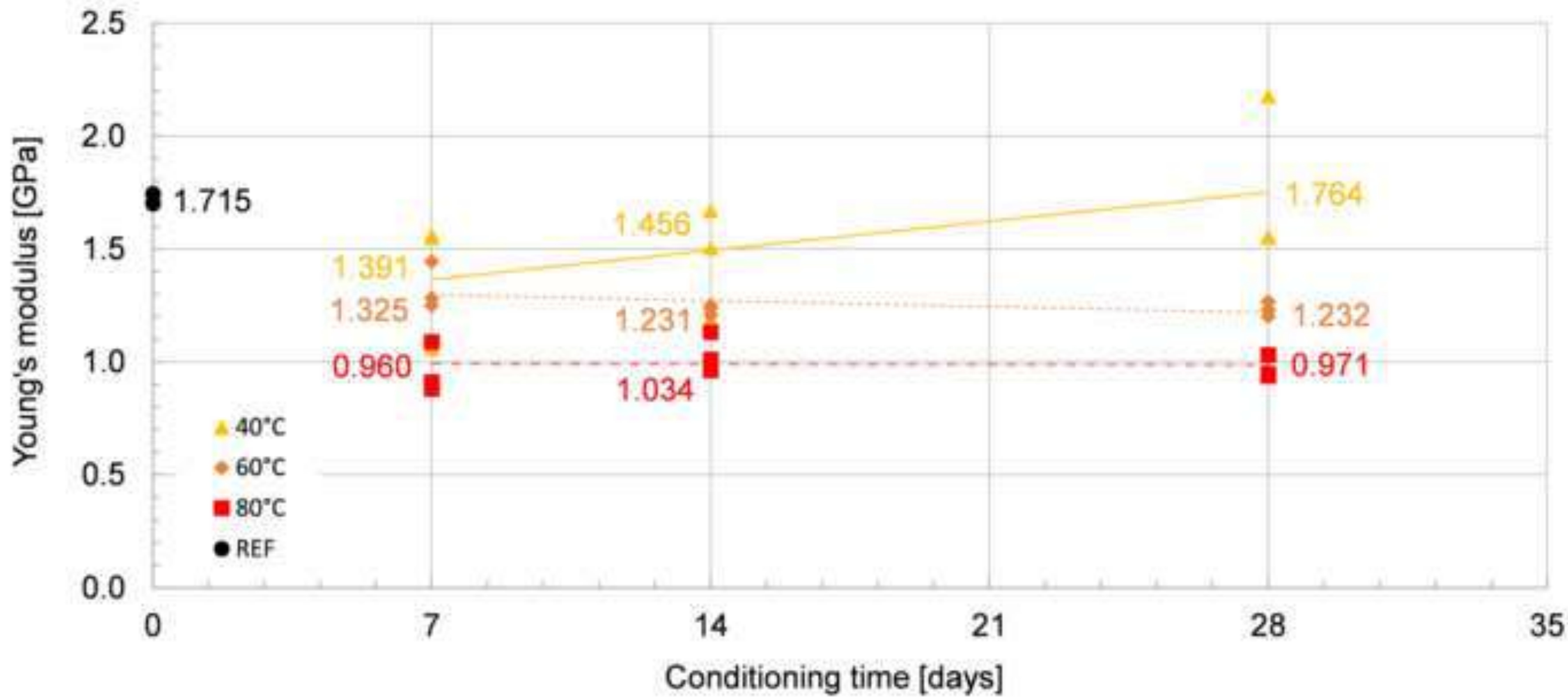


80
75
70
65
60
Shore hardness [SH]
73.6
69.8
71.7
72.6
-20°C; 20°C/45%RH
REF
0
7
14
21
28
35
Conditioning time [days]

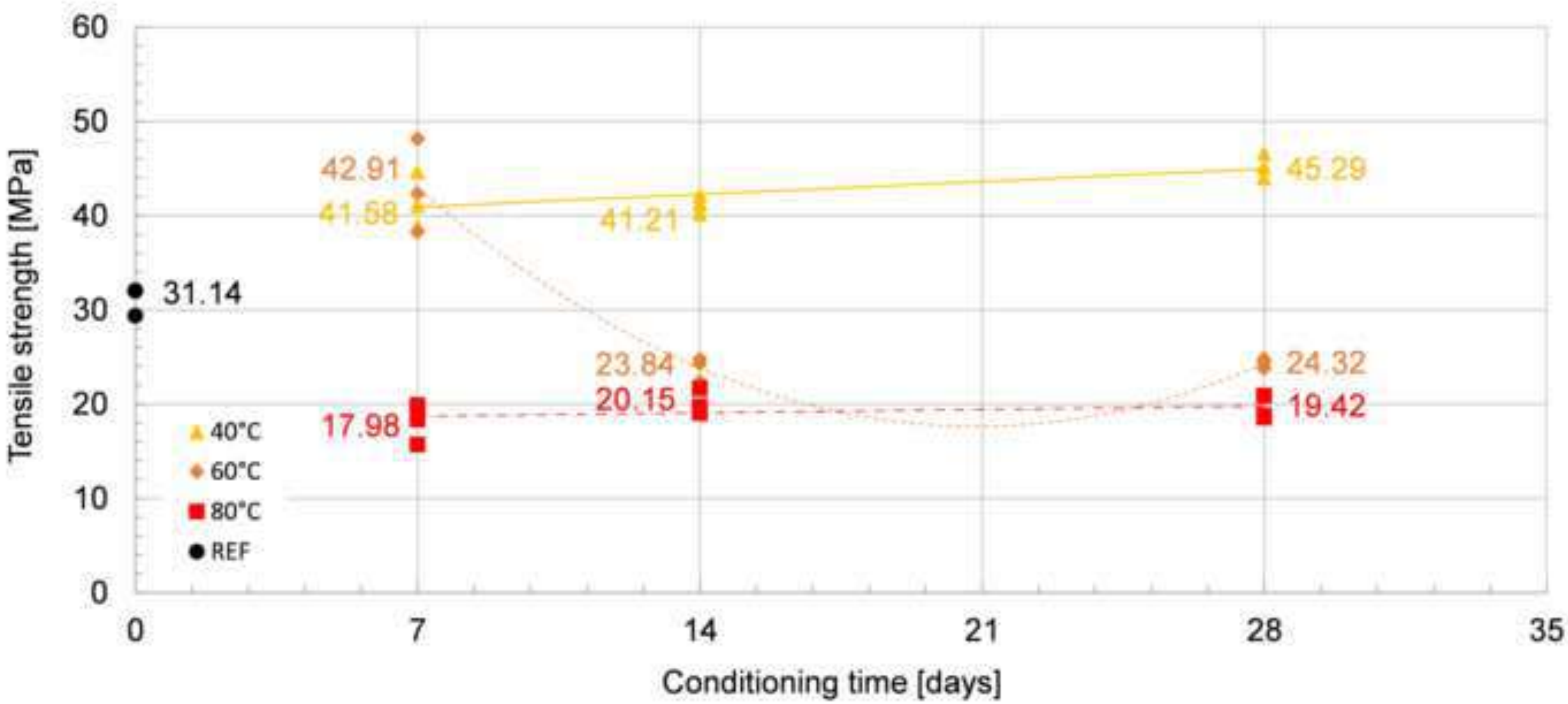

80
75
70
65
60
Shore hardness [SH]
-20°C; 20°C/45%RH
REF
67.5
70.0
67.8
65.2
0
7
14
21
28
35
Conditioning time [days]

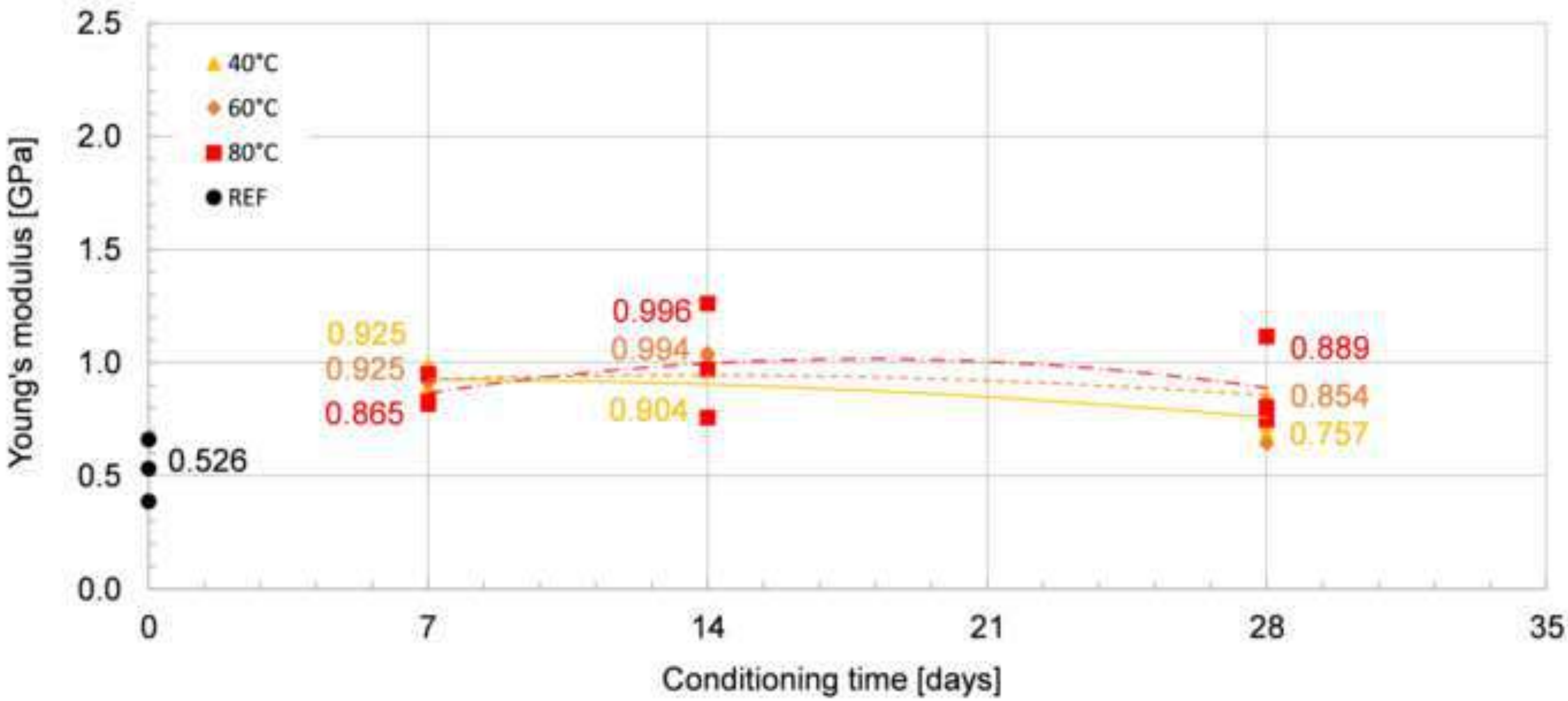

10
16
4
12
12
60
16
3
[mm]

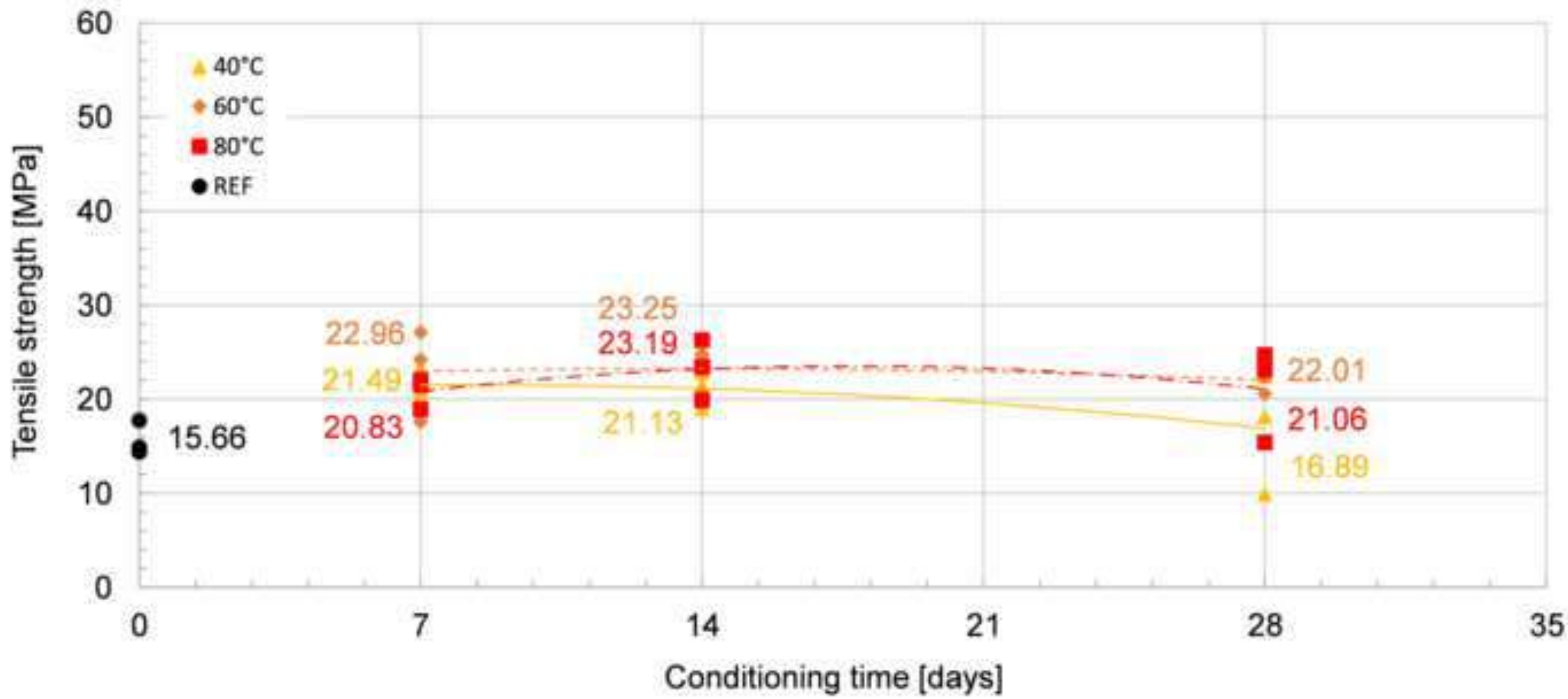
Click here to access/download;Figure;fig5a.png



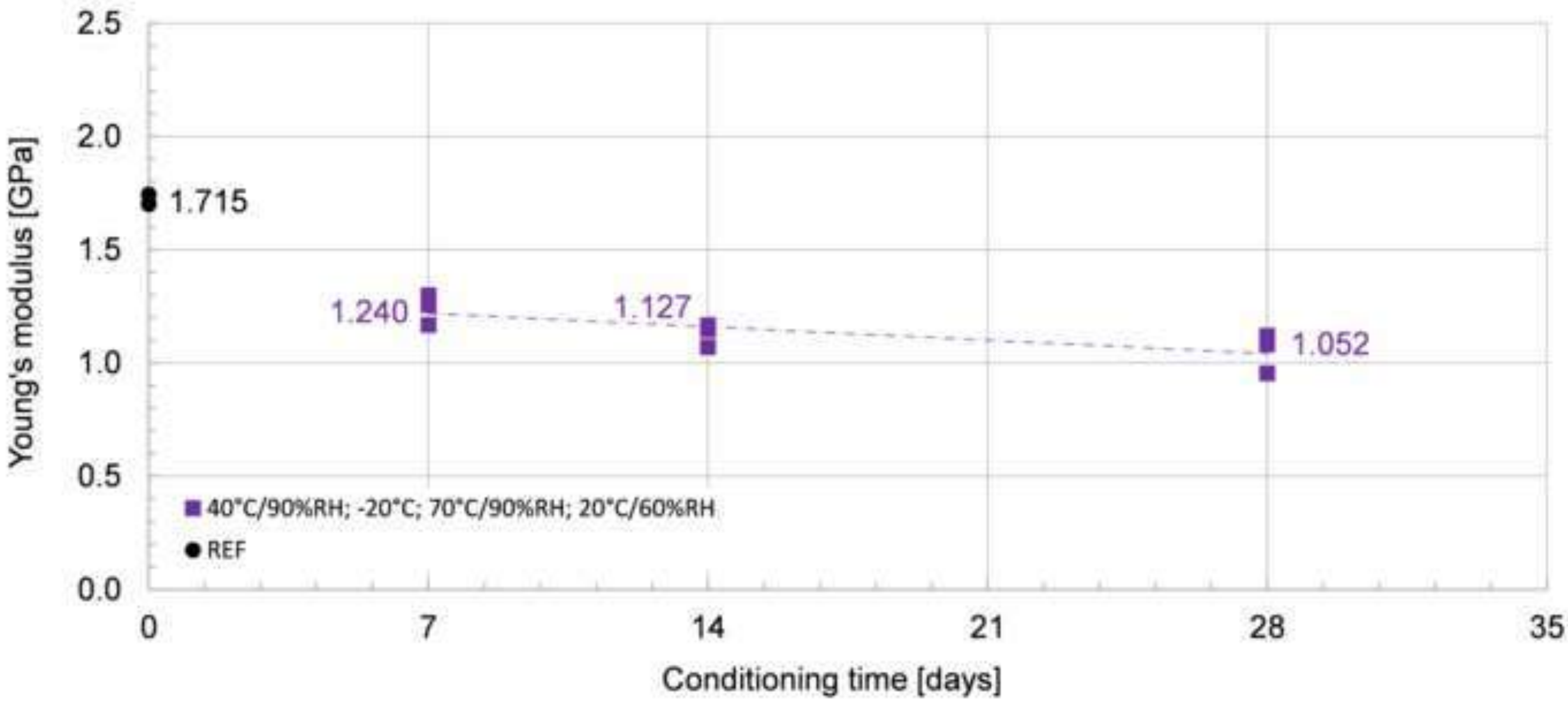
Click here to access/download;Figure;fig5b.png



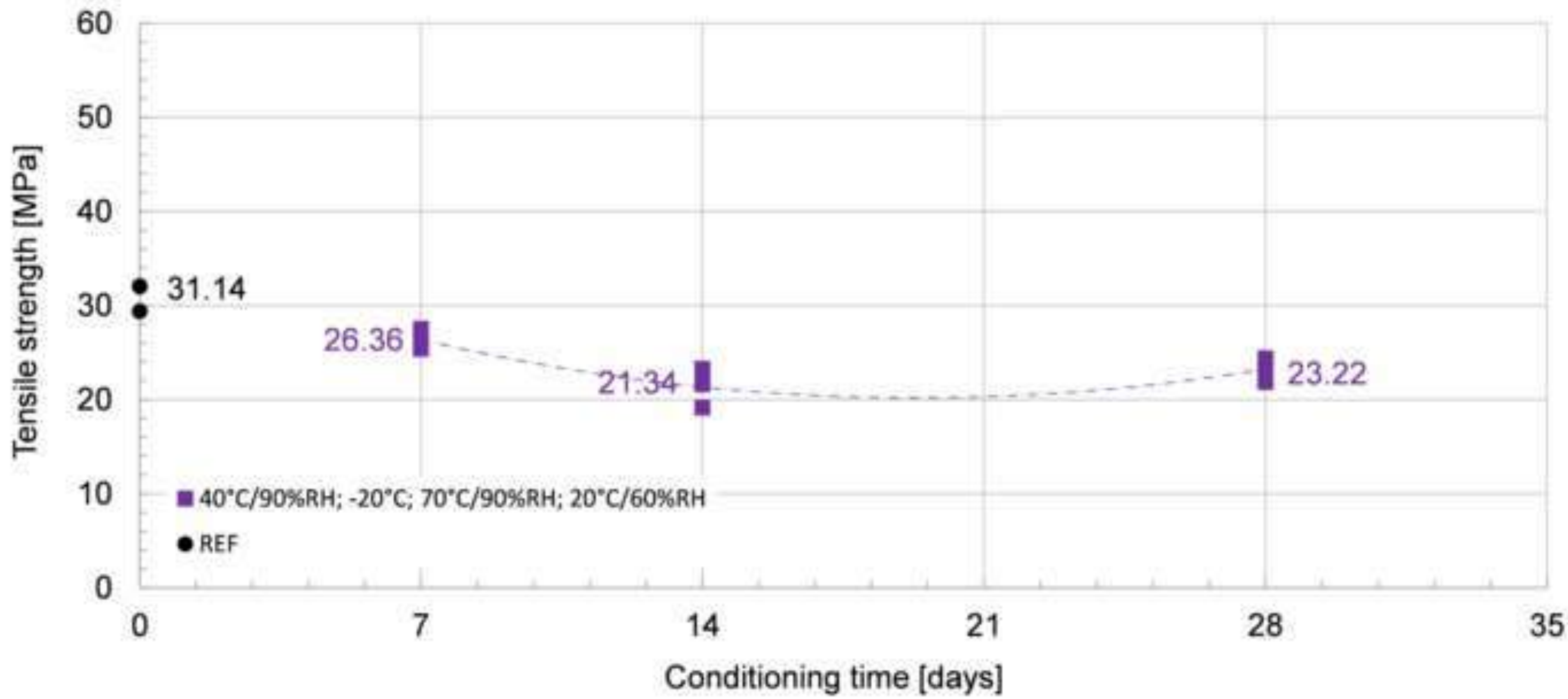

2.5
2.0
1.5
1.0
0.5
0.0
40°C
60°C
80°C
REF
0.925
0.925
0.865
0.996
0.994
0.904
0.889
0.854
0.757
0.526
0
7
14
21
28
35
Young's modulus [GPa]
Conditioning time [days]


60
50
40
30
20
10
0
Tensile strength [MPa]
0
7
14
21
28
35
Conditioning time [days]
40°C
60°C
80°C
REF
15.66
22.96
21.49
20.83
23.25
23.19
21.13
22.01
21.06
16.89

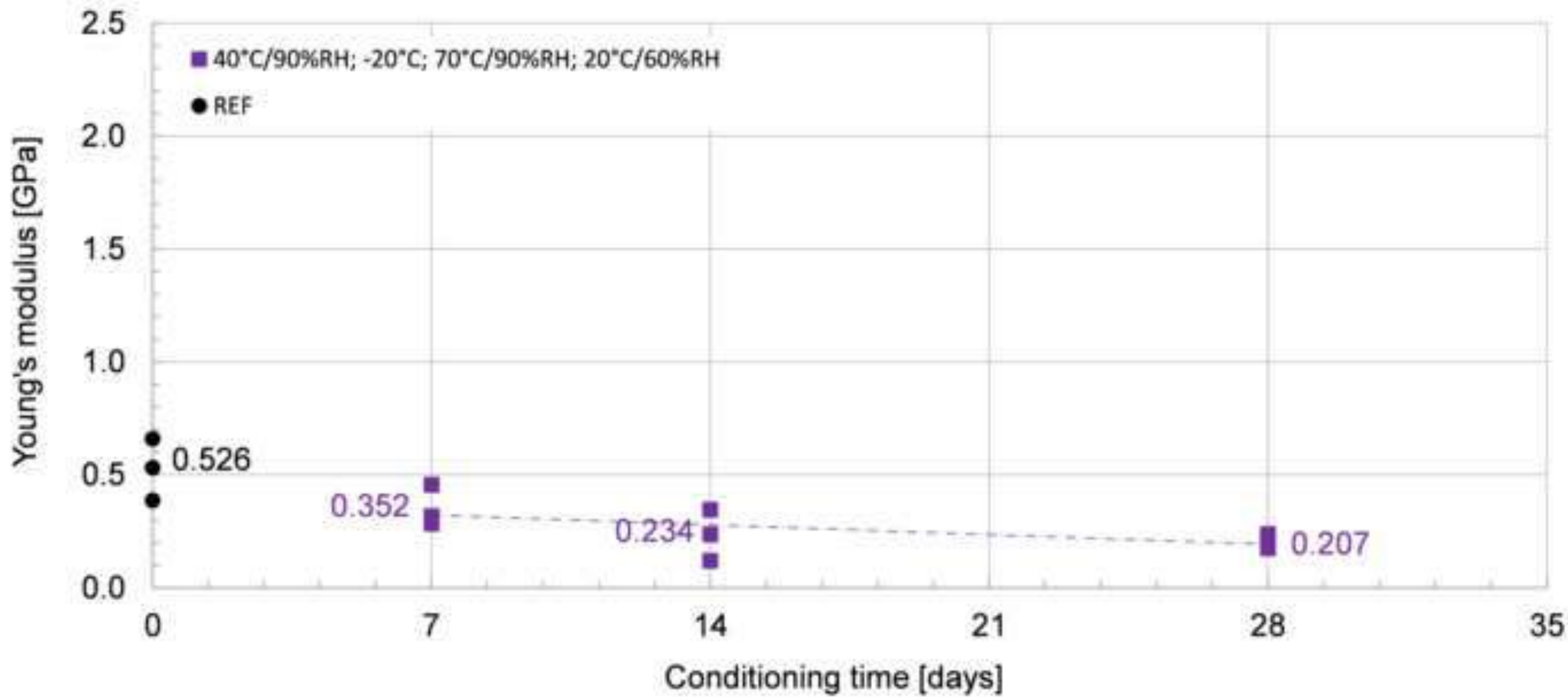
Click here to access/download;Figure;fig7a.png



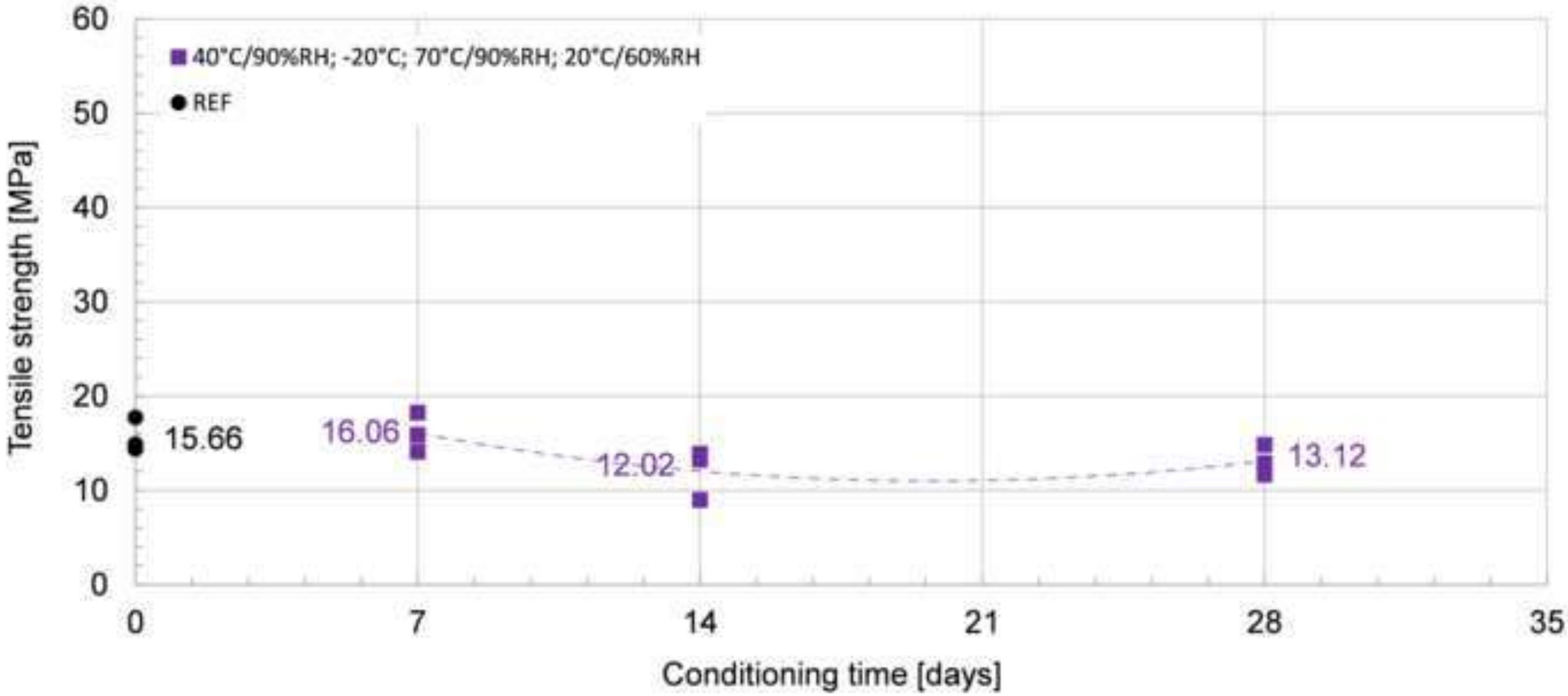
Click here to access/download;Figure;fig7b.png



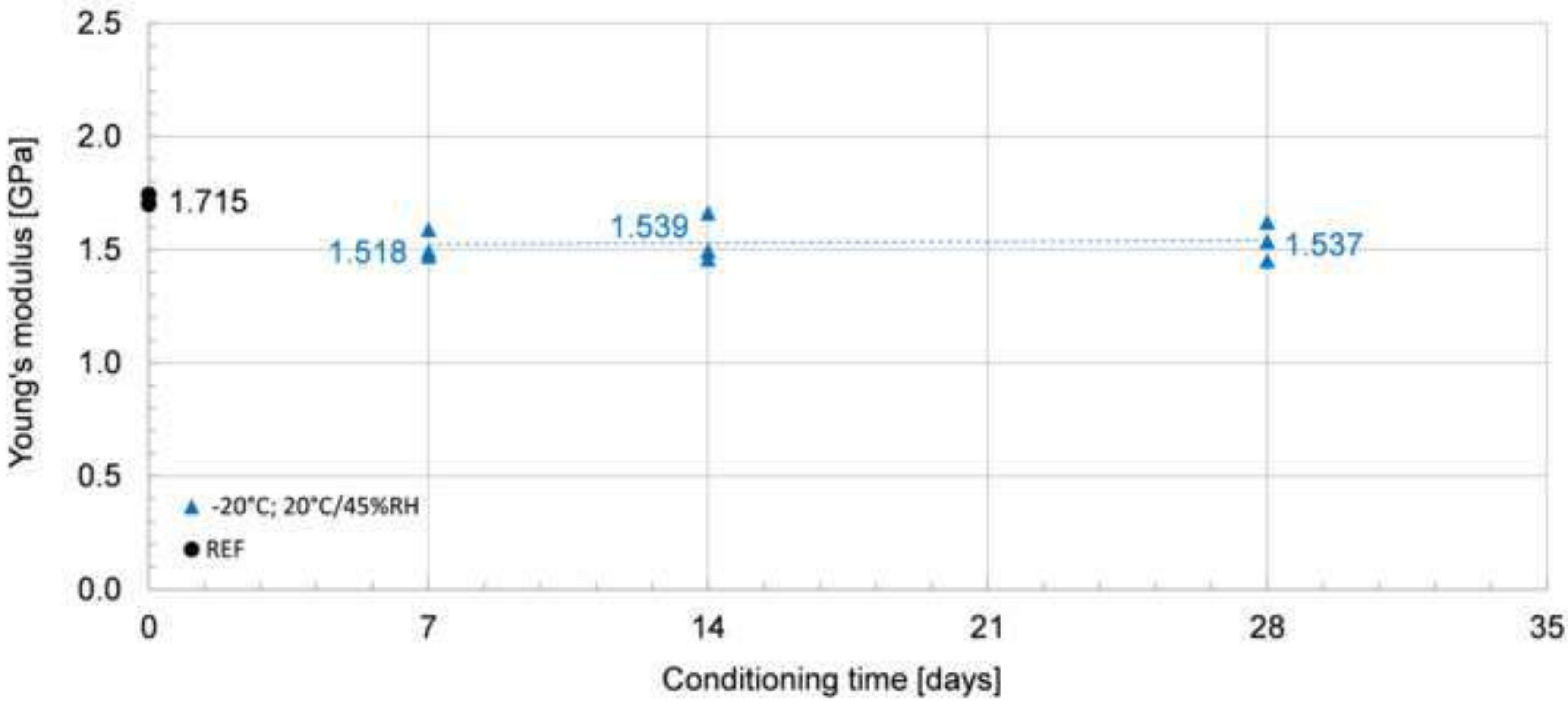
Click here to access/download;Figure;fig8a.png

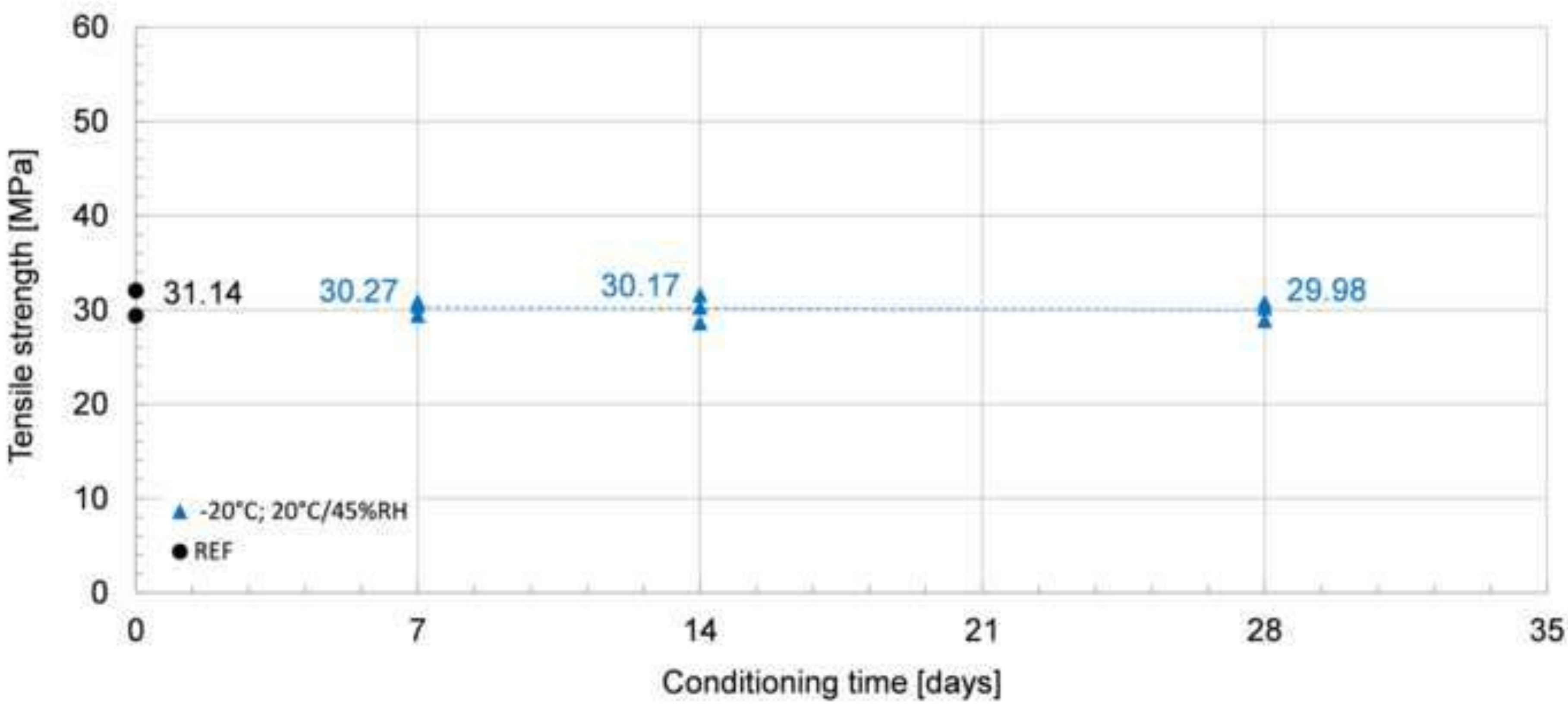


Click here to access/download;Figure;fig8b.png



Click here to access/download;Figure;fig9a.png




60
50
40
30
20
10
0
Tensile strength [MPa]
31.14
30.27
30.17
29.98
-20°C; 20°C/45%RH
REF
0
7
14
21
28
35
Conditioning time [days]