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Al and A356 Alloy Foam Castings Modified with Low Concentrations of Nano-Sized Particles: Structural Study and Compressive Strength Tests

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Abstract: Aluminum and A356 alloy foam castings are produced using a melt-foaming method. Prior to foaming, the melt is modified with nano-sized particles (SiC, TiN, or Al_2O_3). The nano-sized particles are mixed with micro-sized Al particles, which are ultrasonically treated and hot-extruded. Thus, the so-called "modifying nano-composition" is obtained. The resulting compositions are introduced into the melt of the Al foam at the following mass concentrations of nanoparticles: SiC: 0.038 wt. %; TiN: 0.045 wt. %; and Al_2O_3: 0.046 wt. %. For the A356 foam, we use the following concentrations: SiC: 0.039 wt. %; TiN: 0.052 wt. %; and Al_2O_3: 0.086 wt. %. The macrostructure of the foam castings is investigated by CT scanning and 3D analysis. The pore size distributions and accumulative fraction dependencies are determined for all samples. The microstructure of the foam castings is investigated by SEM-EDS analysis. The results confirmed the presence of individual nano-sized particles, as well as clusters of particles in foam walls. The conducted compression tests show a significant increase in the plateau stress (up to 237%) of the modified aluminum foam castings compared to non-modified castings. However, a similar effect of the nano-compositions on A356 alloy foam castings is not observed. The obtained results show that the above-indicated concentrations of nanoparticles can positively influence the mechanical properties of aluminum foam castings. The novelty of the current study is two-fold: (1) such low concentrations of added nanoparticles have never been used before to alter Al foam's properties, and (2) an original method of introducing the nanoparticles into the melt is applied in the form of nano-compositions.

Keywords: Al and Al alloy foam castings; computed tomography (CT) analysis; SEM-EDS analysis; nano-sized particles; compressive strength

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1. Introduction

The use of particles of refractory compounds such as SiC, SiO_2, TiB_2, Al_2O_3, B_4C, etc., to enhance the structure of cell walls and improve the mechanical properties of foam is an area in which researchers work. Three of the existing approaches of enhancing the foam structure and improving the mechanical properties are adding micro-sized particles, adding nano-sized particles, and adding grain refiners. In this Introduction, we will consider such published works.

Micro-composite foams. The authors of [1] investigated a foam obtained by a powder metallurgical method, which contained 8.6% by volume of SiC microparticles, as well as a foam without SiC particles. The results showed that the presence of SiC increases the linear expansion and compressive stress of the foam. It was noted that SiC foam is more brittle than aluminum foam. The influence of the size and content of the SiC microparticles

that were added in a melt on the microstructure and mechanical properties of an AlSi7 alloy foam was investigated in [2]. The foam was obtained by the powder metallurgical method. It was shown that the use of particles with an average size of 3 μm and a content of 3 vol. % led to good foaming and satisfactory mechanical properties. An A356 aluminum alloy melt was foamed, and Al_2O_3 microparticles with volume concentrations of 5, 10, and 15 vol. % were introduced into it. The foams had the same cell sizes. Mechanical tests showed an increase in yield strength, plateau, and Jung's modulus magnitudes with increasing Al_2O_3 particle concentrations [3]. For the production of foam, a ready-made composite, DURALCAN F3S.10S, type metal matrix composite, supplied by A/S Temponik, Denmark, of an aluminum alloy (9% Si, 0.4% Mg) and 10 vol. % of microparticle SiC [4] were used. The composite was melted and foamed with two types of foam extractors: titanium hydride or calcium carbonate. The microstructure and mechanical properties of the resulting foam were investigated. As a parameter for characterizing the microstructure, the ratio D/d was used, where D is the diameter of the test specimen and d is the average size of the foam cells. Data on the foam's porosity are absent, which makes it difficult to compare the obtained data on mechanical properties with the results of other authors. Micro-sized powders of aluminum alloy 6061, TiH_2, and SiC were mixed, homogenized, pressed, sintered at 445 °C, and foamed at 750 °C [5]. It was found that the best foaming of the specimens occurred at low concentrations of SiC—4 wt. %. Only one dependence was presented for the compressive strength of a specimen with a porosity of 40%. Data on the influence of the SiC concentration on strength are absent. Another approach to influence the microstructure of aluminum foams is the addition of aluminum powder in the melt before the foaming [6]. As is known, the surfaces of aluminum particles are covered with a thin layer of aluminum oxide. Thus, oxide particles fell into the melt, which can have an similar effect on the foam to the addition of micro- or nanoparticle Al_2O_3, which is analyzed in this Introduction as well. The authors of [1,2,5] used the same foam preparation method and the same SiC additive, but the foam was made of three different materials, which makes it difficult to compare the obtained results. The general point was that the addition of SiC improved the foam's mechanical properties. The same applies to the other works cited here. All authors used micro-powders and obtained an improvement in foam properties. In general, high concentrations of micro-powders were introduced, and the obtained structure of the foam cell walls was essentially a micro-composite.

Nano-composite foams. Composite foams of aluminum and SiC nanoparticles were obtained by the powder metallurgical method [7]. The examination of experimental specimens' characteristics showed that the introduction of nanoparticles increased their plateau stresses and improved their energy absorption. A comparison was made with aluminum specimens of the same porosity. At a porosity of 60% and with the introduction of 1 vol. % SiC, the plateau stress was 59 MPa, and at a porosity close to 62%, the foam without SiC had a plateau stress of 20 MPa. A reduction in the average cell size (by 50.4%) was also found when the foams were modified with nanoparticles. An original technology for the production of Al, Al-SiC, and Mg foams was presented in [8]. The addition of 10 wt. % nanoparticles of SiC to Al improved the compressive strength by up to 50 MPa before the densification stage compared to the 5 MPa achieved for pure Al. No data were given on the foam's porosity, but the number of pores per inch was provided. Also, an examination of the fracture surface was carried out, showing that the Al foam deforms in a ductile manner. The influence of SiO_2 nanoparticles on the properties of an aluminum alloy foam was investigated in [9]. The nano-composite foam was obtained by melting the Al alloy, introducing the nanoparticles (0.25, 0.5, 0.75, and 1.0 wt. %), ultrasonically stirring the melt, and foaming using TiH_2. The authors found a significant improvement in the mechanical properties of the foam containing nanoparticles. Nano-composite foams reinforced with 0.5 and 0.75 wt. % SiO_2 nanoparticles showed the best foam structure; also, the relative density increased from 0.09 to 0.16, and the plateau stress increased from 0.44 MPa to 1.42 MPa for the Al alloy foam with 0.5 wt. % SiO_2. The Vickers hardness reached its largest increase (78.7 HV) with 0.75 wt. % SiO_2. The authors did not explain why foaming had not

occurred in a large volume of the casting. A ready-made in situ composite Al-4.4Cu-2TiB_2, containing micro-sized TiB_2 particles was foamed in [10]. No data were given on the size of the TiB_2 particles, as well as on their content in the composite material. As a foaming agent, TiH_2 was used. This was the case with the obtained foam containing micro-sized TiB_2 particles. Later, the melt was affected using a powerful ultrasonic device, which led to the fragmentation of TiB_2 microparticles contained in it. The authors proved that using this method, they obtained a nano-composite material and, accordingly, a foam containing nanoparticles. Studies showed higher porosity and higher plateau strength of the foam with nanoparticles. Thus, at a porosity of 82.8%, the plateau stress was 6.7 $\pm$ 2.6 MPa. The authors used FESEM and TEM analysis to prove the existence of nanoparticles. What is interesting here is that X-ray scanning reconstruction was used only in 2D mode, which only gives information on the pore sizes and distribution, as well as on cell width, in one cross-section, which is a disadvantage of this work. An improvement in the mechanical properties of aluminum foam containing 0.75 wt. % SiC nanoparticles was found in [11]. The foam was a part of a new sandwich structure. The authors conducted a separate study of foam only, which is of interest. A friction stir process was used to obtain precursors containing a TiH_2 foaming agent and Al_2O_3 nanoparticles, and an AA5083 alloy was applied as a substrate in [12]. The resulting material was foamed in a laboratory furnace. Samples were obtained from the foam to determine the mechanical properties. A significant increase in plateau stress was achieved compared to similar foams obtained by conventional methods. The value of critical stress was defined by a critical stress curve as 27 MPa. This corresponds to the end of the curve's elastic region. The porosity of the foam was found to be 68%. The authors of [13] produced an aluminum nano-composite—Al + B_4C foam—using the powder metallurgy–Space Holder Technique. The foam had a porosity of 52% and different contents of nanoparticles from 0.5 to 2.5 vol. %. The nano-composite foam with a nanoparticle content of up to 2% had higher strength and yield strength than the aluminum foam with the same porosity. The yield stress increased from 15.95 MPa for the pure aluminum foam to 23.90 MPa at a 2 vol. % B_4C. A very extensive analysis of composite and nano-composite metal foams was carried out in [14]. It was shown that the introduction of micro- and nano-sized particles into the metal of the foam matrix improved its mechanical properties. Articles where the influence of ceramic nanoparticles was studied are relatively sparse. Some difficulties in their application are reported, namely, poor wetting by the melt and nano-sized particles tending to form clusters.

Grain refiners. Another approach was used by a research team to refine the microstructure and improve the mechanical properties of the foamed aluminum alloy A356 [15]. After melting the alloy, they introduced master alloys of Al-5Ti-1B and Al-10Sr into the melt. $BaSO_4$ was used to increase the viscosity of the melt, and $CaCO_3$ was used as a foaming agent. As a result of the used modifiers, the distance between the secondary axes of the dendrites (SDAS) of the cell walls decreased, and the yield and plateau stresses increased. At porosity values of 88%, the plateau stress of the modified foam increased by 47% compared to the unmodified foam. The application of a grain refiner was analyzed as well in [16]. It was shown using thermal analysis and metallography that with the addition of an Al–5Ti–1B master alloy or by increasing the cooling rate of the foam, the aluminum grain size decreased. As is known, a finely divided microstructure increases the strength of alloys.

As can be seen from the studies presented above, typically, nano- or microparticles in concentrations from 0.6 to 10 wt. % were added. The plateau stress depends on the porosity. It is difficult to conduct a good analysis, so we have presented the numerical values of the investigated quantities for each reference. The results stand for different particle types and different porosities, and the data are not always complete. However, it is clear that most often, nano- or micro-SiC particles are introduced into the foam, while there is also a foam containing Al_2O_3 particles. The influence of the nano- and microparticles can be seen in two ways: (1) the grain sizes decrease compared to those of pure aluminum, as some of the particles become centers of crystallization, and (2) the Zenner pinning effect: during

melt crystallization, the nanoparticles prevent movement of the growing grain boundaries by exerting a pinning pressure. In conclusion, it can be argued that the use of micro- and nano-sized particles is a suitable approach to improve the characteristics of aluminum foams. No pre-treatment to improve the wetting of the particles before their introduction into the melt has been reported. There are also no studies with particle concentrations below 0.1 wt. %. In our study, the results of the introduction of nano-sized SiC, TiN, and Al_2O_3 particles on Al foam and A356 aluminum alloy castings are presented. Based on the references cited above, we selected these three types of nanoparticles, which have a proven effect on Al and A356 alloys. To improve the wetting of the particles, a two-step process involving ultrasonic treatment and hot extrusion was used. There are no data indicating that other authors have used such a method for altering the properties of foam castings. The concentration of nano-sized particles in the foam ranged from 0.038 wt. % to 0.086 wt. %. These low concentrations were used in order to establish whether the methodology for low-concentration nanoparticle modification of solid aluminum alloy castings is applicable to foam castings.

2. Materials and Methods

The foam castings of aluminum and the aluminum alloy A356 were investigated. Aluminum and the A356 alloy were produced by Stam Trading JSC, Sofia, Bulgaria. The chemical compositions of Al and A356, obtained using an optical emission spectrometer Q4 Tasman Q101750-C 130, Bruker, (Bruker Belgium SA, Kontich, Belgium) are given in Table 1:

Table 1. Chemical composition of Al and A356.

Element	Si, wt. %	Fe, wt. %	Cu, wt. %	Mg, wt. %	Al
Technical pure Al	0.075	0.103	≤0.0020	0.012	rem.
A356	6.5–7.5	0.487	0.055	0.326	rem.

The added nano-sized particles SiC, Al_2O_3, and TiN were produced by US Research Nanomaterials, Inc. (Houston, TX, USA) with average sizes as follows: SiC: 45 nm, Al_2O_3: 80 nm, TiN: 20 nm; these are taken from producer certificates.

Micro-sized Al particles, which were mixed with nano-sized particles, were produced by Strem Chemicals Inc. (Newburyport, MA, USA) with average size of 31.7 μm, taken from the producer's certificate.

Increasing the melt viscosity was achieved by introducing Ca, which was produced by Alfa Aesar (Thermo Fisher GmbH, Kandel, Germany). TiH_2, which was used as a foaming agent, was manufactured by AG materials Inc. (Taoyuan, Taiwan) with an average particle size of 28.9 μm, measured by Analysette22 NanoTec plus apparatus (Microtrac Inc., York, PA, USA).

A technology for hot extrusion was developed in order to improve the wetting of ceramic nano-sized particles. It involves an ultrasonic treatment of a mixture of nano-sized particles and micro-sized Al particles in a bath of ethyl alcohol, with subsequent drying. The resulting mixture is placed in aluminum containers and heated to a temperature of 500 °C. The container is then placed in the extrusion tool, mounted on a hydraulic press. Under the influence of the applied pressure, the material flows through the nozzle. Wires with a diameter of 4.1 mm were obtained, with the following compositions: SiC + Al, Al_2O_3 + Al, and TiN + Al. The weight ratio of the compositions was 1:3.

The method for obtaining foam castings includes the following stages:

- The melting of 2 kg Al or A356 alloy in a resistance furnace.
- A nano-composition, containing from 0.038 to 0.086 wt. % of nano-sized particles, is introduced at a temperature of 690 °C, and the melt is intensively mixed with an immersed impeller.

- The introduction of 2.5 wt. % Ca into the melt. For Al, the temperature of introduction is 690 °C, and for the A356 alloy, it is 650 °C.
- Intensive stirring to achieve homogenization of Ca in the melt. The stirring speed is 600 rpm for 6 min for both the Al and A356 alloy melts.
- Pouring the melt into a thin-walled metal form with an inner diameter of 130 mm, located in a second resistance furnace heated to 655 °C.
- The introduction of 1.5 wt. % TiH_2 powder.
- Intensive stirring to achieve TiH_2 homogenization in the melt, with a stirring speed of 850 rpm for 90 s for both the Al and A356 alloy melts.
- The decomposition of TiH_2, followed by the separation of H_2 and foaming, where the melt volume increases as a result of pore formation.
- Removing the metal form from the furnace. Cooling of the outer wall using jets of water flowing from a ring located at the top of the metal form. The water temperature is 150 °C, the cooling time is 6 min, and the water flow rate is 3.5 L/min.

Cylindrical foam castings with a diameter of 130 mm and heights of 212 mm for aluminum and for A356 alloy heights of 247 mm were obtained. Samples with dimensions of 18 × 18 × 18 mm^3 were cut from the foam molds. In another work of ours [17], a trend was found for the influence on the samples' porosity regarding their location in the foam casting. It was found that the porosity decreases along the radius from the casting center to the casting surface. In the present work, we investigated samples from the castings with equal or similar porosities.

CT Analysis

Samples of Al and A356 alloy foam castings, modified with nano-compositions, were scanned using an X-ray microtomograph SkyScan 1272, Bruker (Bruker Belgium SA, Kontich, Belgium). The scanning settings were as follows: source voltage 90 kV, source current 111 μA, resolution 2452 × 1640 pxl, image pixel size: 10.885958 μm, rotation step: 0.200°, 360° rotation of sample, filter: Al 0.5 + Cu 0.038. X-ray projection images were processed and reconstructed with NRecon software v.1.7.4.2. CTVox software v. 3.3.1 was used to visualize the obtained model, and CTAn software v.1.23.0.2 was used for processing the reconstructed images and calculation of quantitative data, i.e., the percentage of open, closed, and total porosity, pore diameters, and wall thickness.

SEM-EDSAnalysis

For the SEM examination of the Al foam castings modified with nano-compositions containing TiN and SiC nanoparticles, samples were prepared using the standard grinding and polishing procedures for Al alloys. The samples were analyzed by means of (1) a Quanta 450 FEG SEM by Thermo Fisher Scientific (Thermo Fisher Scientific, Waltham, MA USA), equipped with an energy-dispersive (EDS) X-ray system by AMETEK (used for the sample containing SiC particles—results given in Figure 5) and (2) a JSM-7600F FEG SEM by JEOL (JEOL USA, Inc., Peabody, MA, USA), equipped with an energy-dispersive (EDS) X-ray system by OXFORD instruments (used for the sample containing TiN particles—results given in Figure 6). Both SEM images were taken in secondary electron (SE) mode using an accelerating voltage of 20 kV.

MechanicalCharacterization

Samples from both materials were studied in order to obtain their mechanical properties under quasi-static compression. The experiments were conducted on a servo-hydraulic testing machine, Zwick-Roell HA-250 (ZwickRoell GmbH & Co. KG, Ulm, Germany), at a strain rate of 0.001 s^{-1}.

3. Results and Discussion

Foam castings of aluminum and the aluminum alloy A356, modified with the nano-compositions SiC + Al, Al_2O_3 + Al, and TiN + Al, were obtained and investigated. The mass concentrations of nanoparticles in the melt are shown in Table 2.

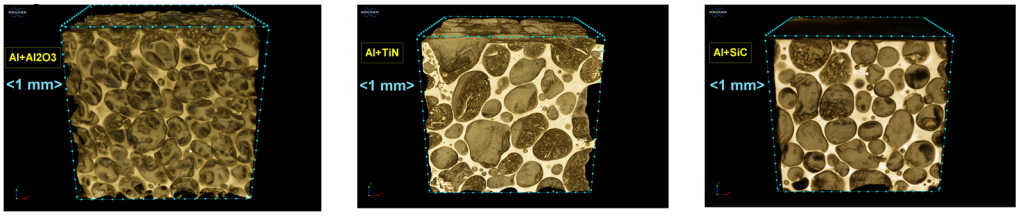
Table 2. Mass concentrations of nanoparticles in the melt.

Nano-Sized Particles	Concentration in Al, wt. %	Concentration in A356, wt. %
SiC	0.038	0.039
TiN	0.045	0.052
Al_2O_3	0.046	0.086

It can be seen from Table 2 that the mass concentrations of nanoparticles obtained here are much lower than the concentrations of nano- or microparticles reported by other authors (Section 1). The main purpose of the nanoparticles here is to act as additional crystallization centers and thus positively influence the properties of the foam. Our previous studies on the modification of aluminum alloy castings have proven the effectiveness of nanoparticles at similar concentrations [18].

3.1. CT Analysis

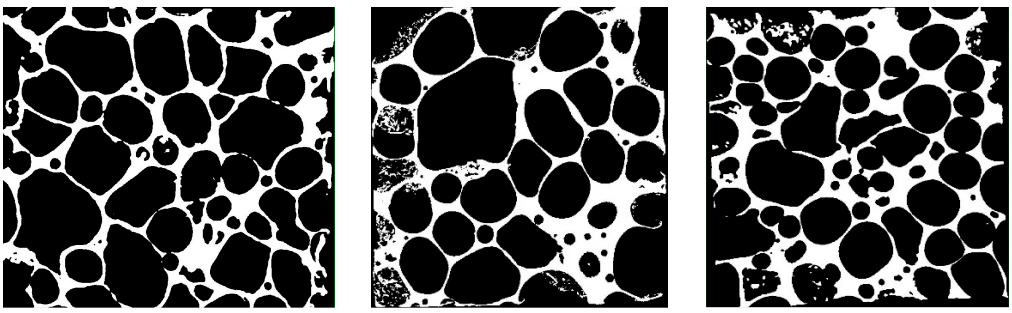
The CTVox software visualizations of foam casting samples modified with nano-compositions are given in Figure 1. A scale bar is provided in the upper left corner of the images.



(**a**) Al+ Al_2O_3 (**b**) Al+ TiN (**c**) Al + SiC

Figure 1. CTVox visualizations of modified Al foam castings.

If we look at Figure 1, it can be seen that sample Al + SiC is the most rounded and regular in the shape (close to a circle) of its pores. The pores of sample Al + Al_2O_3 look shallower compared to the two other samples. Analogous visualizations were created for all examined samples. In Figure 2, the binarized 2D images, which are processed with the software CTAn v.1.23.0.2, are given in a section that is perpendicular to the cross-sections shown in Figure 1 of the same samples. In this way, the entire volume is processed, and the calculated parameters are given in Table 3.



(**a**) Al+ Al_2O_3 (**b**) Al+ TiN (**c**) Al + SiC

Figure 2. Two-dimensional binarized images of modified Al foam castings; the size of the sections is 18 × 18 mm^2.

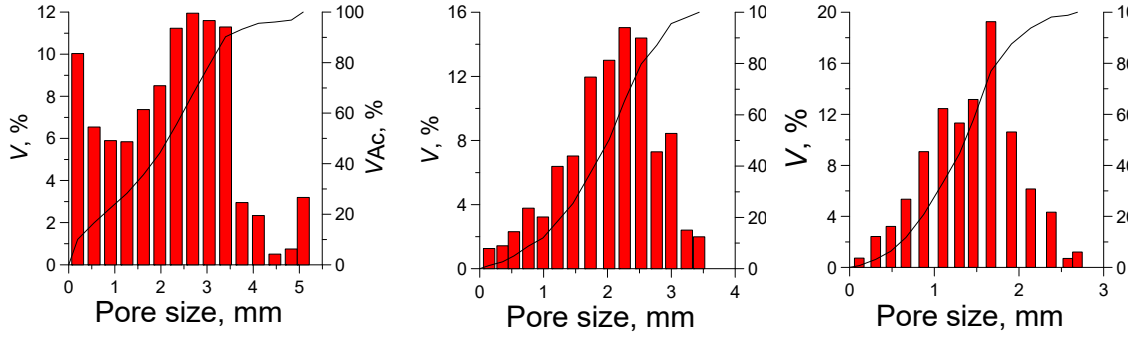
As can be seen in Figures 1 and 2, in Al foam castings with TiN (Figures 1b and 2b) and with SiC (Figures 1c and 2c), smaller particles are present in the walls as well as in the pores. These particles could be clusters of nanoparticles or chemical compounds of Ca or Ti. Also, the presence of pores with relatively small diameters is registered in Figure 2b,c.

The 3D CT analysis data are given in Table 3.

Table 3. Three-dimensional CT analysis results.

Sample	Closed Porosity, %	Open Porosity, %	Total Porosity, %	Average Diameter of Pores, mm	Average Thickness of Walls, mm
Al/SiC	0.39	74.2	74.3	1.77 ± 0.74	0.73 ± 0.28
Al/SiC	0.71	80.1	80.2	2.90 ± 1.48	0.84 ± 0.31
Al/SiC	0.50	76.2	76.3	2.06 ± 0.96	0.86 ± 0.30
Al/TiN	2.20	78.2	78.6	2.82 ± 1.51	0.66 ± 0.29
Al/TiN	3.13	76.7	77.4	2.66 ± 1.30	0.71 ± 0.29
Al/TiN	2.12	73.7	74.2	2.59 ± 1.25	0.81 ± 0.28
Al/Al_2O_3	0.15	79.4	79.4	1.38 ± 0.53	0.47 ± 0.13
Al/Al_2O_3	0.14	79.9	79.9	1.75 ± 0.62	0.45 ± 0.15
Al/Al_2O_3	0.36	81.9	82	1.75 ± 0.77	0.54 ± 0.18
A356/SiC	0.82	79.3	79.5	1.72 ± 0.75	0.42 ± 0.09
A356/SiC	0.91	78.2	78.4	1.81 ± 0.76	0.47 ± 0.13
A356/TiN	0.64	82.4	82.5	2.96 ± 0.85	0.42 ± 0.12
A356/TiN	0.39	85.4	85.5	1.73 ± 0.71	0.32 ± 0.09
A356/Al_2O_3	0.66	90.2	90.2	2.59 ± 1.21	0.50 ± 0.11
A356/Al_2O_3	0.84	84.7	84.8	2.12 ± 0.97	0.66 ± 0.37

According to the CT analysis data (Table 3), the nanoparticle-modified Al and alloy A356 foam samples have mostly open porosities and very low percentages of closed porosities. The porosity of the modified Al samples varies from 74.3% to 80%, the average pore diameter varies from (1.38 ± 0.53) mm to (2.90 ± 1.48) mm, and the average pore wall thickness varies from (0.47 ± 0.13) mm to (0.86 ± 0.30) mm. The average diameter of the pores and the average thickness of the walls of Al foams, modified with Al_2O_3, are significantly lower compared to SiC/TiN-modified foams. In our opinion, this difference is related to the spherical shape of the Al_2O_3 nanoparticles and their influence on the foam's structure. However, further investigations are needed to fully understand and explain this phenomenon. In the A356 alloy samples, the porosity ranges from 78.4% to 90.2%, the average pore diameter changes from (1.72 ± 0.75) mm to (2.96 ± 0.85) mm, and the average pore wall thickness varies from (0.32 ± 0.09) mm to (0.66 ± 0.37) mm. Figure 3a–c show the pore size distributions and accumulation fraction dependences of Al foams for samples modified with the three types of nanoparticles.



(**a**) Al foam modified with Al+TiN (**b**) Al foam modified with Al+SiC (**c**) Al foam modified with Al+Al_2O_3

Figure 3. Pore size distributions and accumulation fraction dependences of Al foam samples modified with different types of nanoparticles.

Figure 4 shows a comparison of the accumulative fraction V_{Ac} for three Al samples modified with nano-sized particles and an Al sample without modification, obtained in our previous work [17].

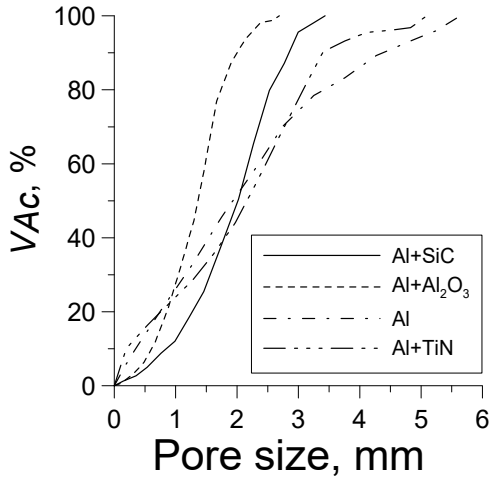


Figure 4. Comparison of the accumulative fraction V_{Ac} for three Al samples modified with nano-sized particles and an Al sample without modification Adapted from Ref. [17].

The derivatives (slope angle) of the V_{Ac} functions for SiC + Al and Al_2O_3 + Al have greater values than the derivatives of V_{Ac} for Al and for TiN + Al. As is known, this parameter characterizes the homogeneity of the foam. The higher its value is, the greater the foam's homogeneity is [19]. Therefore, it can be concluded that to some extent, nanoparticles improve the homogeneity of the foam.

Furthermore, a decrease in the mean pore diameter variation range of the nanoparticle-modified foams was observed compared to the mean pore diameter range of the Al foam. For example, the mean range of the Al foam varied from (1.32 ± 0.58) to (2.88 ± 1.48) mm [17], and for Al foams modified with Al_2O_3, it ranged from (1.38 ± 0.53) to (1.75 ± 0.77) mm.

The authors of [8] conducted measurements of Al and Mg foams using FESEM. Their results are as follows: pore sizes vary between 100 µm and 500 µm, and cell width varies between 50 µm and 100 µm. The relative density of foams (ρ/ρs) in [9] was defined as the ratio of the apparent density of the foams (ρ) to the fully dense composites (ρs). This method does not provide information on all foam parameters. In [12], image processing software was used to determine the porosity. As can be seen from the brief analysis, not all the macroscopic parameters of the foam are determined using the above methods. In our opinion, the use of CT analysis, which was applied here, gives much wider possibilities for evaluating the characteristics of a foam.

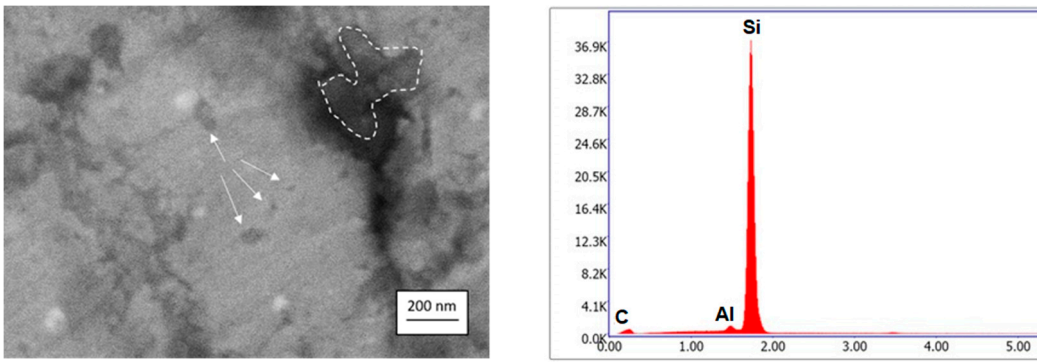
3.2. SEM-EDS Analysis

SEM-EDS analysis of the microstructure of the Al foam samples containing SiC and TiN nanoparticles was carried out. Figure 5 shows an SEM image of a foam containing SiC (a) and the EDS analysis result (spectrum) for a SiC nano-sized particle.

In Figure 5a, individual SiC nanoparticles can be observed (marked with arrows), as well as an agglomeration of particles in a crack (dashed area). The individual particle sizes vary between approximately 20 and 100 nm, while the cluster size is around 400 nm.

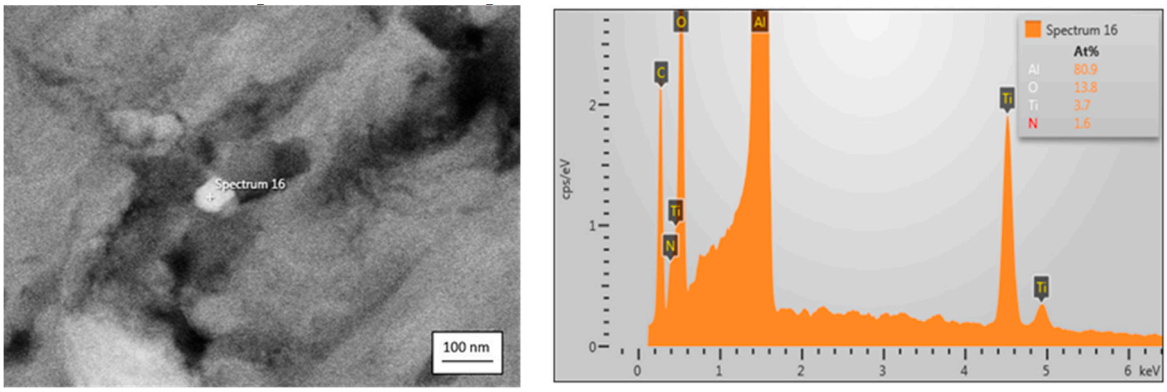
Figure 6 shows a SEM image of a foam containing TiN (a) and the EDS analysis result (spectrum) for a TiN particle.

An individual TiN nanoparticle with a size of 30 to 40 nm is observed. EDS analysis proved the presence of titanium and nitrogen. Aluminum and oxygen were also present. In our opinion, this is due to the pre-treatment of the nanoparticle, involving hot extrusion of a mixture of aluminum microparticles and TiN nanoparticles. Aluminum particles are usually oxidized. As a result, the formation of a thin layer of aluminum on the particles and the presence of oxygen from the oxide layer is possible.



(**a**) (**b**)

Figure 5. SEM image in SE mode (**a**) and EDS analysis result for SiC particle (**b**) of Al foam sample, modified with SiC.



(**a**) (**b**)

Figure 6. SEM image in SE mode (**a**) and EDS analysis of TiN particle (marked as "Spectrum 16") (**b**) of Al foam sample, modified with TiN.

Similar results for foam microstructure were published in [20], where the nano-compositions of Al and 5 vol. % of SiC or Al_2O_3 nanoparticles were investigated. Agglomerations of nanoparticles with sizes between 1 and 5 µm were observed. It was determined that an Al/SiC nano-composition exhibits a higher degree of agglomeration compared to an Al/Al_2O_3 nano-composition.

3.3. Mechanical Characterization

Before analyzing the results of the mechanical properties of the nanoparticle-modified foam samples, we will briefly summarize our results on $\overline{\sigma_{pl}}$ = F (Porosity) [17], as well as those obtained by other authors. $\overline{\sigma_{pl}}$ is defined as the arithmetic mean of σ_{20} and σ_{30}, measured by compression testing of the foam samples. Such comparisons for Al foam and A356 alloy foam are shown in Figure 7a,b. The linear dependences are based on our data [17], and the points are the data of other authors [7,19,21–26]. It can be seen that there is a difference in the magnitude of $\overline{\sigma_{pl}}$. Therefore, it can be concluded that not only the porosity affects the mechanical properties. In our opinion, the method for obtaining the foam and the parameters of the foaming process, such as the temperature, concentration and type of foaming agent, viscosity of the melt, etc., probably matter. The compression test results for the Al and A356 foam samples modified with SiC, Al_2O_3, and TiN are presented in Table 4. The values of σ_{20}, σ_{30}, $\overline{\sigma_{pl}}$, and the compressive strength (σ_{60}) of the modified Al foam decrease with increasing porosity. The same dependence was observed for the modified A356 foam. This result would be more convincing with a larger range of porosity variation, but the aim of this research is mainly to prove the effect of modifying the foam with relatively low concentrations of nanoparticles.

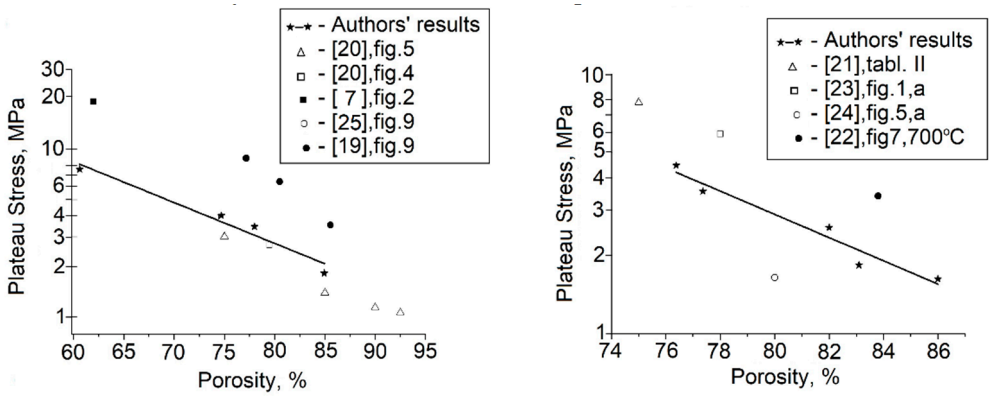


Figure 7. Comparison of plateau stress $\overline{\sigma_{pl}}$ from the authors' results, Adapted from Ref. [17] and those of other authors for (**a**) Al foam Adapted from Refs. [7,17,19,21,26], (**b**) A356 alloy foam Adapted from Refs. [17,22–25].

Table 4. Compressive property results for Al foam castings and A356 alloy foam castings, modified with different types of nanoparticles.

Sample	Porosity, %	σ (20 %), MPa	σ (30 %), MPa	$\overline{\sigma_{pl}}$, MPa	Compressive Strength, MPa
Al/SiC	74.3	13.11	14.01	13.6	37.0
Al/SiC	80.2	3.64	5.44	4.03	15.6
Al/SiC	76.3	9.34	11.31	3.46	30.7
Al/TiN	78.6	4.94	6.80	5.87	18.3
Al/TiN	77.4	4.93	5.82	5.37	19.8
Al/TiN	74.2	8.45	8.66	8.56	24.5
Al/Al_2O_3	79.4	4.53	5.42	4.96	13.2
Al/Al_2O_3	79.9	4.62	5.32	5.00	12.4
Al/Al_2O_3	82.0	4.13	5.07	4.80	11.8
A356/SiC	79.5	2.71	2.72	2.71	5.4
A356/SiC	78.4	3.01	4.37	3.68	12.8
A356/TiN	82.5	2.96	2.57	2.75	4.3
A356/TiN	85.5	0.85	1.17	1.00	4.3
A356/Al_2O_3	90.2	0.82	0.8	0.81	1.0
A356/Al_2O_3	84.8	3.27	2.18	2.75	4.1

In Figure 8, the compressive stress–strain curves of the modified and unmodified Al foams are shown, and the tested specimens before and after compression are presented in Table 5. The modification is carried out by the addition of SiC, Al_2O_3, and TiN nanoparticles. The stress–strain curves for Al foams were obtained in our previous investigation [17] according to the methodology described in the present work.

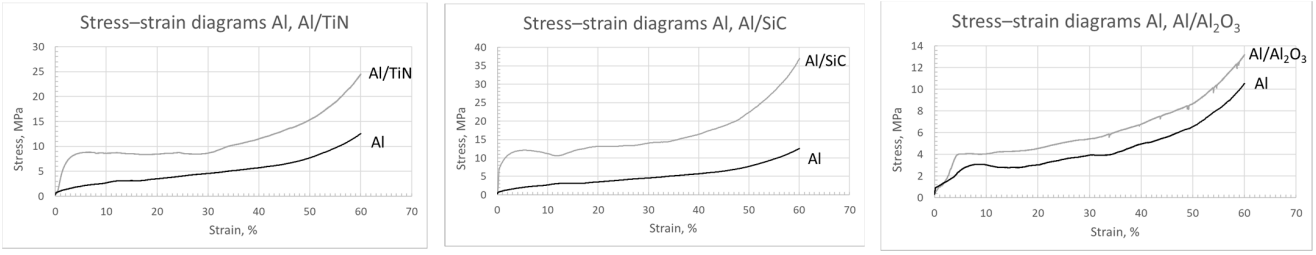
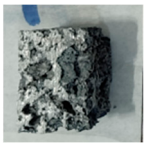
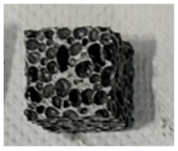
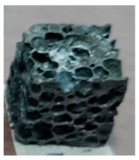
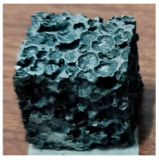
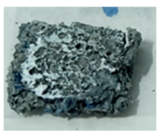
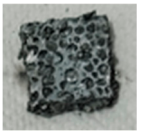
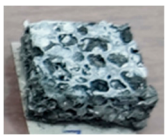


Figure 8. Compressive stress–strain curves of Al foam castings modified with nanoparticles with concentrations of 0.045 wt. % TiN (**a**), 0.038 wt. % SiC (**b**), and 0.046 wt. % Al_2O_3 (**c**). Curves for unmodified Al foam castings are taken from the authors' results in Adapted from Ref. [17].

Table 5. Tested specimens before and after compression.

Sample	Al	Al/SiC	Al/TiN	Al/Al_2O_3
Before compression				
After compression				

All stress–strain curves shown are characterized by three distinct regions: the elastic region with linear–elastic deformation due to the low strain, the plastic plateau with almost constant stresses, and the densification region, characterized by the crush of the cell walls. A step-by-step analysis (Figure 9a) of the fracture process shows that at the beginning of the compression test, the cell walls start to bend and the linear–elastic deformation is homogeneous throughout the specimen (up to 5% strain—Figure 9b). After reaching the first maximum in stress (3–5% strain), the deformation starts to localize (Figure 9c), and the cells begin to collapse by plastic yielding (Figure 9e) due to the formation of plastic hinges (Figure 9d) at the section of the maximum bent moment. It is obvious that within the localized band, the collapse of the cells is governed by an asymmetric shearing mode of deformation, while away from the band, the deformation continues to be symmetric and homogeneous. With the strain increase, the deformation spreads (Figure 9f) in the adjacent rows of cells and also becomes asymmetric. The collapse ends (Figure 9g) when the opposing cell walls or their broken fragments start to touch each other and eventually pack together, leading to the densification of the pore material and a rapid increase in its stiffness (Figure 9h).

The effect of the modification is clearly visible. All the curves of the modified foam have higher stress values than those of the unmodified foam. At the same time, the porosities of the compared samples are similar. The Al + TiN foam casting sample has a porosity of 74.2%, and the Al + SiC foam casting sample has a porosity of 74.3%. They are compared with the Al foam casting sample, which has a porosity of 74.7%. The Al + Al_2O_3 foam casting sample has a porosity of 79.4% and is compared to an Al foam casting sample with a porosity of 78.0%. The increase in plateau strength for the Al + TiN sample is 112%, for Al + SiC, it is 237%, and for Al + Al_2O_3, it is 43.7 %. It can also be seen from the results that the level of the plateau stress increases with the porosity decreasing. Another comparison of the magnitude of the plateau was made for all samples modified with nanoparticles with linear dependences: $\overline{\sigma_{pl}}$ = F (Porosity) for the Al foam casting samples (Figure 10a) and the A356 alloy foam casting samples (Figure 10b), based on our data presented in [17]. In Figure 10 a, it can be seen that all the modified foam casting samples have higher $\overline{\sigma_{pl}}$s than those of the pure Al foam casting samples. The graph for alloy A356 shows no apparent effect of modification with nanoparticles on the magnitude of $\overline{\sigma_{pl}}$. This may be due to the alloying elements Si, Mg, etc., that are present in this alloy, as well as the added Ca and Ti that are necessary for foaming. As a result, nanoparticles with the above-mentioned concentrations are not effective enough to positively influence the plateau stress of A356 alloy foam castings. Of course, extensive research is needed to clarify this experimental fact.

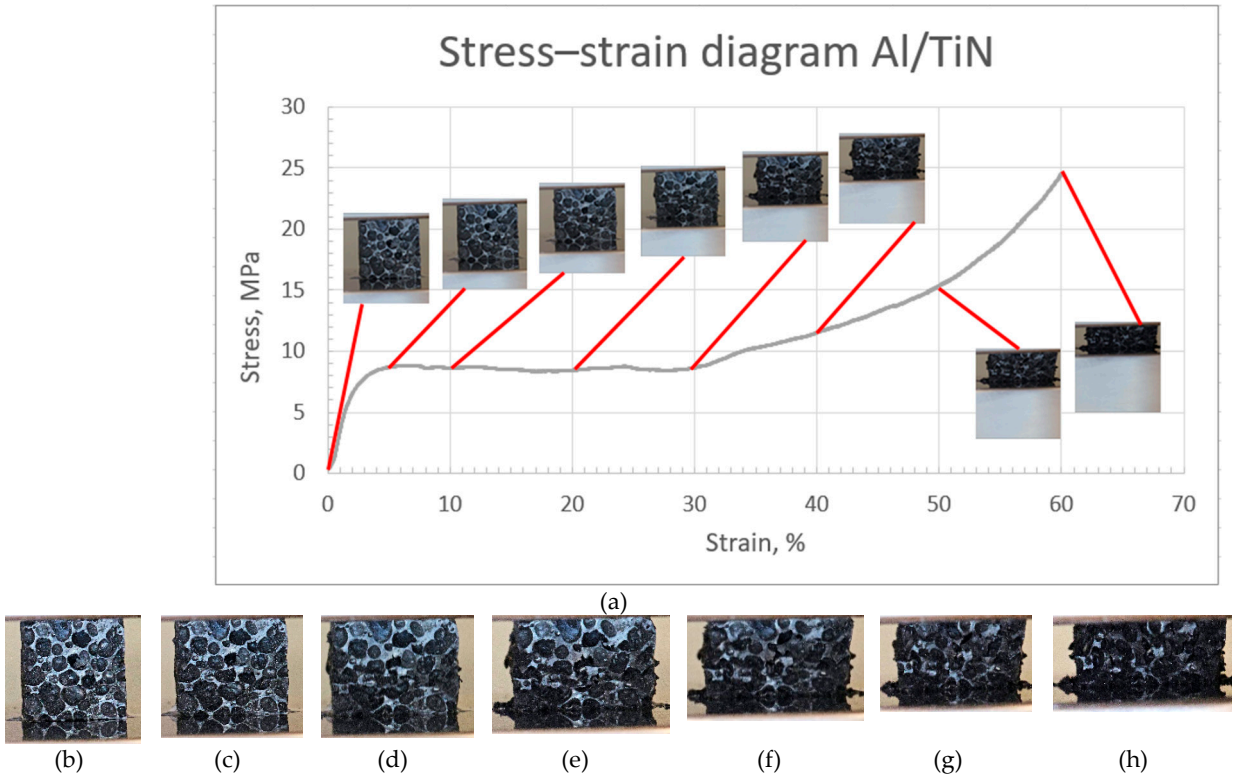


Figure 9. Macroscopic fracture of Al foam castings modified with 0.045 wt. % TiN nanoparticles: (**a**) stress–strain diagram and sample macrostructure at different strains, as follows: (**b**) ε = 0%; (**c**) ε = 10%; (**d**) ε = 20%; (**e**) ε = 30%; (**f**) ε = 40%; (**g**) ε = 50%; (**h**) ε = 60%.

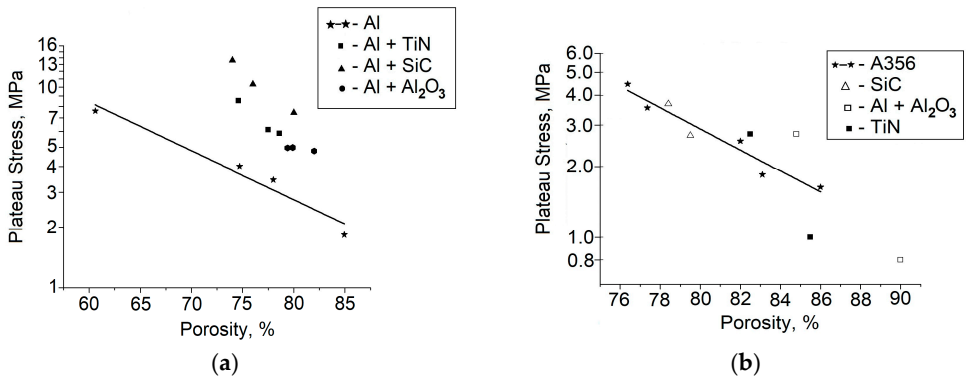


Figure 10. Dependence of plateau stress $\overline{\sigma_{pl}}$ on the foam porosity (**a**) for unmodified Al foam casting Adapted from Ref. [17] and indicated by points for Al foam castings, modified with different nanoparticles; (**b**) for unmodified A356 alloy foam casting Adapted from Ref. [17] and indicated by points for those modified with different nanoparticles of A356 alloy foam castings.

Low-porosity materials of Al and SiC nanoparticles, with concentrations of 2.8 wt. % and 6 wt. %, were obtained [27]. Mechanical tests showed that an increase in the nanoparticle concentration led to an increase in the plateau stress, yet the increase was small—from 9.2 to 10.3 MPa compared to that of SiC, which was 2.8 and a 6 wt. % concentration. We cannot make a comparison with our results due to a lack of data on the samples' porosity. But still, the value of the plateau stress is close to our data for the sample with a porosity of 75% and SiC concentration of 0.038 wt. %.

A comparison was made of the plateau stress, obtained in our work, with the result of a previously published article [28] for an Al-SiC composite with a porosity of 75% and a SiC concentration of 10 vol. %. The values are, respectively, 13.56 MPa at a concentration of

0.038 wt. % of SiC nanoparticles and 8.59 MPa at a concentration of SiC microparticles of 10 vol. %.

The plateau stress for an aluminum composite containing 3 vol. % SiC [29] and with a porosity of 75.8% is 21 MPa compared to our data mentioned above. It can be seen that our data values are lower than those in [29]. This once again suggests that porosity alone is not sufficient to compare the mechanical properties of foams. Other dimensions characterizing the foam, such as pore sizes, wall thickness, etc., must also be taken into account.

In [30], in fig.20, data from six different authors on the dependence of σp on the porosity of an aluminum foam were shown. At a relative density of 0.125, the value obtained by different authors varied from 1.5 MPa to 4.5 MPa. These data showed the difficulty that researchers have in summarizing and comparing experimental data. Similar conclusions were drawn by the authors of [14] about an aluminum foam that was reinforced with micro-sized and nano-sized ceramic particles. Micro-sized ceramic particles stabilized bubbles by increasing the viscosity, preventing bubbles from coalescing and inhibiting their growth. A high content of particles, located in the cell wall, gave the foam a level of fragility. Nano-sized ceramic particles reduced the foam's brittleness compared to micro-sized ones. In addition, small additions of nanoparticles improved the foam structure, refined the pores, ensured the homogeneity of the pore distribution, and refined the foam walls' microstructures, which led to an increase in mechanical properties. The dependence of yield stress on the foam's relative density was compared to unmodified foam. An increase was observed as a result of the introduction of particles. A comparison with other authors was not made, but the authors drew the conclusion that because of the diverse manufacturing and testing conditions used in different studies, detailed evolution trends of foams' properties as a function of a given experimental variable can hardly be expressed. For drawing such conclusions, the conditions, such as the influence of particle size on foaming behavior and mechanical properties, should be strictly controlled.

In [31], the plastic collapse of an AA7075 alloy foam reinforced with micro-sized SiC and TiB2 is compared with an A356 alloy foam containing micro-sized SiC particles, which are, respectively, 25MPa and 5MPa (Figure 12b). Here, again, it was difficult to analyze the obtained results, because the alloys are different, as are the introduced micro-powders.

In order to improve the mechanical properties of Al foam, technologies with different concentrations of nanoparticles in the matrix are being developed, as each researcher adopts a different foam reinforcement strategy. Thus, in [12], the concentration of the used Al_2O_3 nanoparticles is 2 wt. %, in [8], the concentration of SiC nanoparticles is 10 wt. %, in [11], the concentration of SiC is 0.75 wt. %, and in [9], the concentration of SiO_2 is 0.75 wt. %. Based on scientific works that are known to us, the lower limit of the used nanoparticles concentration is 0.75 wt. %. Our previous research [18] shows that at nanoparticle concentrations of maximum 0.1 wt. %, the mechanical characteristics of aluminum alloy castings can be improved. Here, this is also confirmed for Al foam castings. Moreover, this result is valid for three types of nanoparticles: SiC, Al_2O_3, and TiN. In our opinion, at low concentrations of nanoparticles, they act as additional centers of crystallization. Thus, the microstructure of the cell walls is refined and, as a consequence, the mechanical properties of the foam increase. At high concentrations of nano-sized particles, which is typical for the composites, the applied force is distributed by both the metal matrix and the nanoparticles due to the significant amount of these in the matrix. This mechanism is different from the one described above, where the mechanical properties increase as a result of the refinement of the microstructure. In our opinion, the current research gives a perspective for improving the mechanical properties of foam materials not only with an aluminum matrix, but also with matrices of other metals.

4. Conclusions

- Foam castings were produced by foaming melts of Al and the A356 alloy, and then modified with compositions containing SiC, Al_2O_3, or TiN nano-sized parti-

cles. The mass concentrations of nanoparticles in the foam ranged from 0.038 wt. % to 0.086 wt. %.

- The foam castings' macrostructure is investigated by CT scanning and 3D analysis. The distribution and accumulative fraction dependencies of the pore samples were determined for all samples. As a result, samples' porosities are determined, as well as the pore diameters and widths of walls within the sample volume. It was established that the above-indicated parameters of the modified foams do not differ significantly from the ones of unmodified foams. Only the cumulative curves for the Al foam casting samples modified with SiC and/or Al_2O_3 show better pore homogeneity compared to pure Al foam casting samples.
- The foam casting's microstructure is investigated by SEM-EDS analysis. The results proved the presence of individual nano-sized particles, as well as clusters of particles in foam walls.
- The quasi-static test results show that the compressive strength of the modified Al foam casting samples have higher values than the unmodified ones. The plateau stress of the modified foam increases by 237% for the modification with a SiC composition, by 112% for a TiN composition, and by 43.7% for an Al_2O_3 composition.
- The modification of A356 alloy foam castings with concentrations of nano-sized particles below 0.1 wt. % does not affect the mechanical properties of the foam. This is probably related to the presence of alloying elements and the higher level of brittleness of the foam, as recorded by other authors.
- The conducted comparative analysis of the plateau stress of pure aluminum foam castings and A356 alloy foam castings depending on the porosity, according to data obtained by various authors, including our data, cannot be summarized in a general relationship.
- The novelty of the current study is two-fold: (1) such low concentrations of added nanoparticles have been never used before to alter Al foam's properties, and (2) for the first time, nanoparticles are being introduced into the melt in the form of nano-compositions.

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

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