MATERIAL SCIENCE

Effect of Periwinkle Shell Ash Reinforcement on the Mechanical Performance of Stir-Cast Zinc Alloy Composites

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Abstract

The mechanical response of zinc alloy composites made from locally available shell ash feedstock by stir casting technique was explored and studied with varying shell ash content of periwinkle. Shells were calcined at temperatures more than 600 °C, milled and sieved through 250 µm mesh before incorporation in the alloy for 10 different compositions from 0 to 5 wt%. Thirty cast rods were used in the experiment and maximum compressive stress, tensile stress, percentage elongation and impact response were reported. The increase in compressive stress from 75.73 MPa for the unreinforced alloy to 83.92 MPa at 2 wt% ash (10.8%) was followed by a sharp decrease with increasing level of ash content. The elongation of the sample was 14.92% at 2 wt% while the control was 1.96%. The tensile behaviour was different: unreinforced specimen was 69.23 MPa and the highest value for the reinforced specimen was 43.85 MPa with 4 wt% reinforcement. Thus, the tested additions were seen to improve some of the selected deformation parameters but not all those increase capacities for tensile strength. The incompatibility of the reported numerical scales for the impact chart and written summary prohibited comparisons regarding impact energy until later when the sources were verified. Note also that this was the original record; no attempt was made to assign individual replicate values or uncertainty estimates so that no statistical significance could be claimed or a definite explanation for the variations at that time could be made. Respective findings illustrate compositional trade-offs of mechanical properties and establish criteria for verifying, before proceeding to engineering or biomedical assessment, a reproducible formulation.

Keywords: compressive strength; periwinkle shell ash; stir casting; tensile strength; zinc alloy composites

1 Introduction

Applications where a combination of strength, ductility and chemical behaviour is required are still of interest for zinc-based materials. Zinc alloys have been of special interest in the biomedical field due to the fact that the degradation rate of zinc can be more controllable than some other biodegradable metals. However, mechanical performance is still a key constraint and the processing-microstructure-mechanical behaviour relationship is not simple [1, 2]. Reinforcement is one way to alter the characteristics of cast metals. It is not only the reinforcement content that is important for its success, but also the way the particles are incorporated, distributed and bonded within the matrix. The tensile response reported for zinc alloys is dependent on the structural condition and deformation mechanisms, and high elongation does not necessarily mean that the material is a good load-bearing material [3]. Therefore, the performance of any new zinc-based composite should be reported in

more than one mechanical parameter. The shells of the periwinkle, which are usually discarded after eating in the coastal areas of Nigeria, have been studied as low cost fillers for composites. Umunakwe et al. [4] reported on the mechanical properties of periwinkle-shell-reinforced aluminium 6063, and Adah et al. [5] reinforced recycled polymers with shell derived material. Olorunfemi et al. [6] then investigated a hybrid rice-husk-ash/periwinkle-shell-ash reinforcement for recycled aluminium composites. The results of these studies demonstrate the practical interest of the shell derived particulates, but cannot be directly applied to a nominal zinc-alloy casting system. A recent study of the ZA-27 alloy reinforced with periwinkle shells was conducted under acidic exposure and showed that the size of the particles and the amount of loading are important to the response of this system [7]. The present experimental record is concerned with a different question: what happens to the compressive stress, tensile stress and elongation of a cast alloy with varying ash content from 0 to 5 wt% when prepared separately? The difference is important because a seemingly beneficial level of reinforcement for one property can decrease another. The purpose of this paper was then to arrange and critically evaluate the existing mechanical data for the 10 reinforcement levels and to compare each of these with the unreinforced control and to determine which compositions should be studied further. The analysis was intentionally restricted to the observations recorded in the project record. Where data were not provided, no microscopy, corrosion testing, statistical significance testing or biological validation was inferred.

2 Related work

2.1 Mechanical performance of zinc-based materials

In the other, Chen et al. [1] outlined the opportunities and the current challenges in practice with zinc alloys for biodegradable internal fixation. Similarly, Kong et al. [2] have reported that mechanical properties of zinc-based implants are strongly influenced by the alloy chemistry and the manufacturing process. While this takes place, it is important to note that these observations do not constitute suitability criteria for a newly cast material. The reactivity to corrosion, release of ions and biological response would still need to be investigated separately. Huang et al. [3] highlighted the strain softening and the difference between fracture elongation and uniform elongation in biodegradable zinc alloys. The analysis is relevant here, because the percentage elongation must not be considered as evidence of a favourable structural performance of a material when a significant percentage is reached. The tensile strength should be looked at in combination with deformation to failure and repeatability.

2.2 Shell-derived reinforcements in composite production

Periwinkle-shell particulate/aluminium composites were prepared by Umunakwe et al. [4] and they found that the mechanical properties of the composites were influenced by the size of the particulates and the processing conditions. They were also able to do some work on the microstructural evaluation, so they could talk a little bit more about particle distribution. The primary difference between the present invention and the prior art lies in the use of a zinc alloy matrix and the relatively restricted range of amount of ash present in relatively small amount and in a relatively fine joining between the elements. Adah et al. [5] recycled the pericarp of ash and provided mechanical properties of the composition. Aluminium with ash reinforcement has also been used to obtain ash reinforcement in recycled aluminium, which Olorunfemi et al. [6] reported the favourable changes in its strength. Generally the direction of change indicated was positive, although for some the behavior

was dependent on matrix and the casting and reinforcement preparation was inconsistent making any optimum for content of shells unacceptable.

2.3 Specific research gap and contribution

Bala et al. [7] investigated ZA-27 composites containing 2-10 wt% of shells and various particle-size fractions; they focused on corrosion resistance in 0.5 M hydrochloric acid. The mechanical data available, on the other hand, is obtained as a mechanical dataset of ten reinforcement levels from 0 to 5 wt% with tensile, compressive and elongation responses. The principal contribution is the recognition of the absence of any common optimum over these properties, as well as the large discontinuities which do not have an explanation that is merely a function of composition. It is not a proven statement that zinc alloys always become stronger when they are mixed with ash.

3 Materials and methodology

3.1 Materials and preparation of the reinforcement

The materials used in the reinforcement and how they are prepared. The experimental material used was an alloy supplied as a zinc alloy by the local vendor and periwinkle shells collected from a market in the South eastern part of Nigeria, Ihiagwa. The shells were washed and dried prior to the calcination in an open hearth furnace at a temperature of more than 600 °C and then ground to a powder, which was sieved through a 250 μm sieve. The additional conditioning step was taken at the Federal University of Technology, Akure, foundry workshop, but the specific temperature and residence time for the additional conditioning step were not recorded.



(a) Shell calcination (b) Cast rod specimens

Figure 1. Shell calcination and examples of fabricated rod specimens, reproduced from the original experimental record.

3.2 Composite fabrication and composition levels

The casting route reported was melting the base alloy in a furnace, adding the desired ash charge, stirring, and pouring into a sand mould of the desired shape to make the rod. The write-up mentions preheating to about 419 °C which is close to the melting point of zinc, but does not mention the actual holding temperature, stirring speed, mixing time or cooling rate. Therefore, the casting process may be termed with the term "stir casting" but it will not be possible to replicate exactly because of the missing settings. Ten nominal reinforcement conditions were considered: 0, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5 and 5.0 wt%. In total, three rods were prepared for each condition, totaling 30 rods of about

150 mm in total length. It did not include specimen-by-specimen test record that indicated which rods were used for each test. The numbers of preparations were thus not used as independent replicate measurements for statistical analysis.

3.3 Mechanical testing

The initial experimental programme was comprised of impact testing, compression testing and tensile testing. Elongation to fracture was also obtained from the tensile tests. Numerical density results were not kept in the results section of the method and were not used in the present analysis, although this method mentioned density was measured using Archimedes principle. No test standard were used, no dimensions of the gauge were given, no model of the testing machine, no speed of the cross-head and no impact-test geometry were detailed which makes comparisons with published standardised measurements difficult.

3.4 Data transcription and descriptive comparisons

The three internally consistent mechanical-property charts in the source report were transcribed at the ten specified reinforcement levels. The resulting values were compared with their respective unreinforced controls. The relative change in any property, R, was calculated as

$$\Delta R(\%) = \frac{R_i - R_0}{R_0} \times 100 \quad (1)$$

where R_0 is the chart value at 0 wt% ash and R_i the chart value of the selected reinforced composition. The difference in points is only reported, and the uncertainty and statistical significance are not estimated. No optimal or fitted response surface was employed and no raw level test observations/variance estimates were provided. A Source Data Audit revealed notice of Impact energy conflict. On an axis labelled joules, the numbers 32.50–38.76 joules appeared on the source impact graph, and in the abstract and concluding bottommost summary it was found that the control and 2.5 wt% each had 3.97 J. Since these two numerical scales were not harmonised from the available documentations no quantitative comparison of the impacts was provided, but rather a series of impacts (no quantitative comparison between impacts can be provided). This restriction is imposed prior to the results interpretation.

4 Results and discussion

4.1 Overview of measured mechanical response

Table 1. Chart-reported mechanical results for the ten reinforcement compositions.

Ash (wt%)	Compression (MPa)	Tension (MPa)	Elongation (%)
0	75.73	69.23	1.96
1	72.99	19.82	2.66
1.5	68.59	16.75	4.33
2	83.92	32.63	14.92
2.5	42.99	38.52	9.96
3	5.32	33.88	2.84
3.5	23.33	10.95	6.88

Ash (wt%)	Compression (MPa)	Tension (MPa)	Elongation (%)
4	44.09	43.85	4.05
4.5	58.03	35.06	1.71
5	45.82	32.47	2.33

Table 1 shows the main measurements taken. There were no predictable monotonic relationships between the composition of the ash and the three responses. The first peak (compressive) was distinct at 2wt% and the second peak (tensile) was in the unreinforced alloy. The greatest elongation was obtained at 2 wt%. These differences illustrate the necessity that needs to be faced with the implementation of a single label: the only possible average is "improved mechanical performance" which should be assessed on each of its own scale, and its own scale of control.

4.2 Compressive response

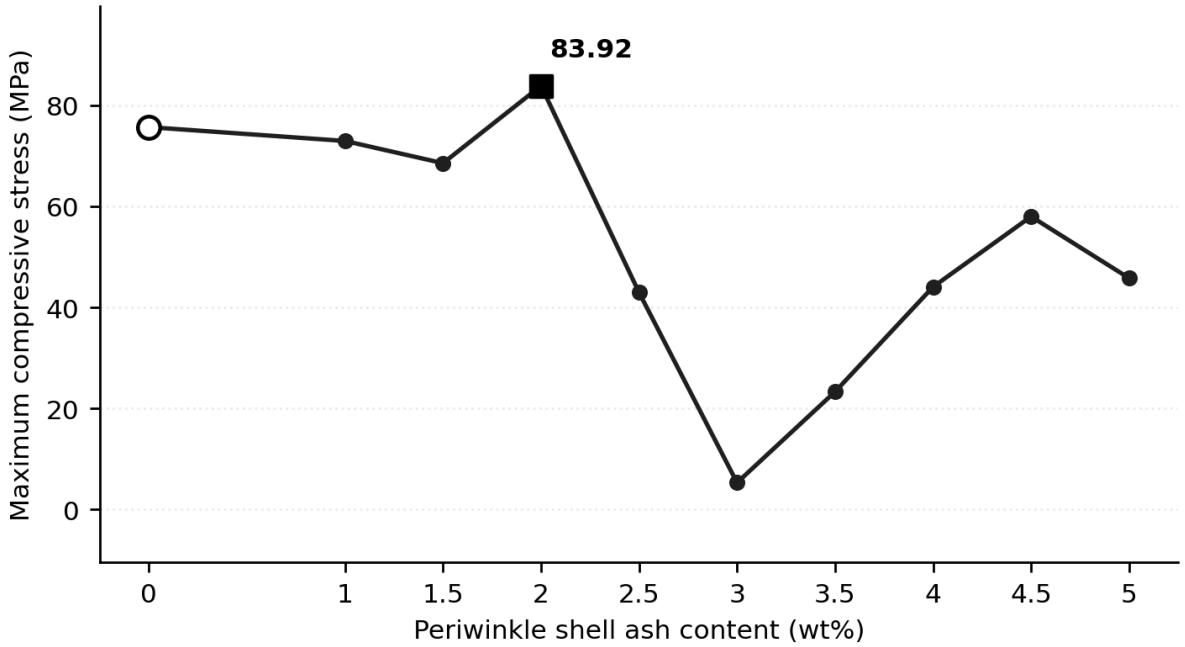

Figure 2. Maximum compressive stress against periwinkle shell ash content, transcribed from the project results chart.

The maximum compressive stress of the control specimen (without reinforcement) was 75.73 MPa. Values at 1 and 1.5 wt% were lower, at 72.99 and 68.59 MPa, respectively. The 2 wt% composition had the highest value of 83.92 MPa, which is 10.8% higher than the control. It was found that this peak was not sustained; the peak value of the second composition (2.5 wt%) decreased to 42.99 MPa and the 3 wt% sample ended up being reported as 5.32 MPa (Figure 2). The very low figure at 3 wt% is noteworthy. It may be because of casting defects, uneven distribution of reinforcement, dimensional or test problems and/or a genuine weakening response. No density measurements, fracture photographs, microscopy results or repeat-test results are recorded in the present record, to differentiate between these explanations. But, particle agglomeration is too early to say is a proven cause. Recovery to 58.03 MPa at 4.5 wt% and 45.82 MPa at 5 wt% further suggests that the outcome is not true a linear function of reinforcement loading.

4.3 Tensile response

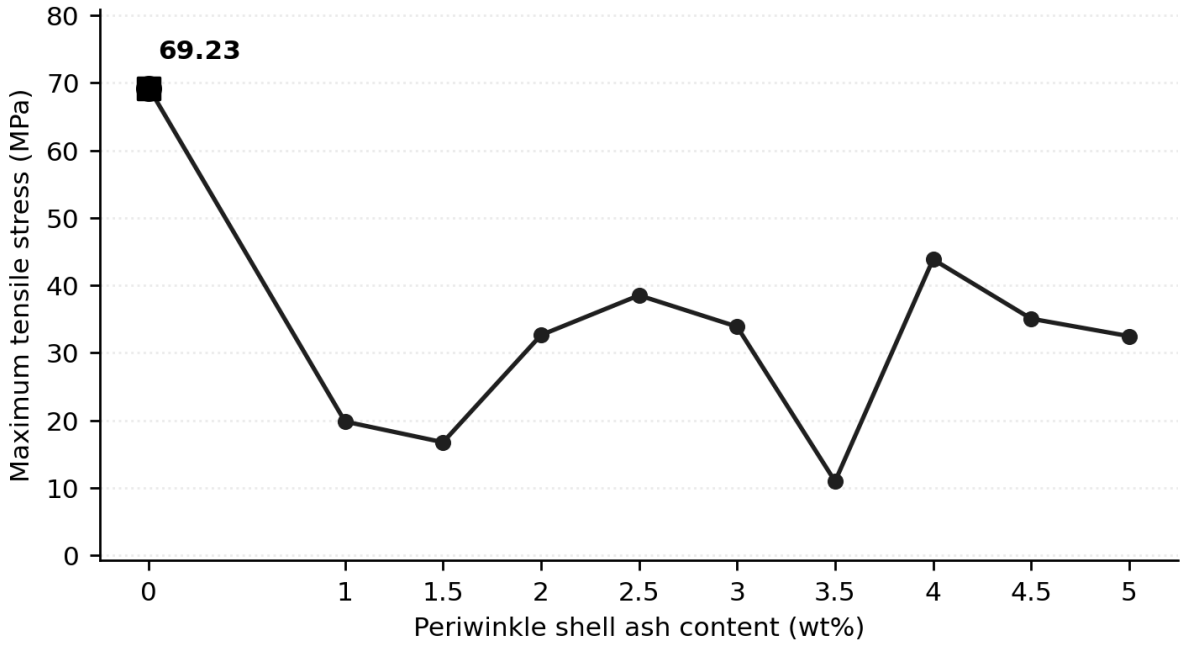

Figure 3. Maximum tensile stress against periwinkle shell ash content, transcribed from the project results chart.

The tensile response was another action than the compression response. The maximum tensile stress was reported for unreinforced material (69.23 MPa). When added ash content was 1wt% and 1.5wt%, they decreased 19.82 and 16.75MPa, respectively. The values were then restored to 32.63 MPa at 2 wt% and 38.52 MPa at 2.5 wt%, and then dropped back to 10.95 MPa at 3.5 wt%. The maximum value obtained for reinforced compositions from Figure 3 is 43.85MPa for 4wt% which corresponds to 36.7 % decrease from the unreinforced control value. The correction is made to eliminate an incorrect inference drawn in the original abstract of the project that 38.52 MPa @ 2.5 wt% was the best tensile outcome. This did not correspond to the largest tension value nor the best of the reinforced specimens in the graph. The most basic null hypothesis to be rejected is that the addition of ash led to increases in tensile strength of the base alloy. A series of changes in the color of the photo from the beginning to the end can be because of many factors of reinforcement and casting quality but without the structural analysis and individual test traces it is not possible to decide which.

4.4 Ductility assessed from percentage elongation

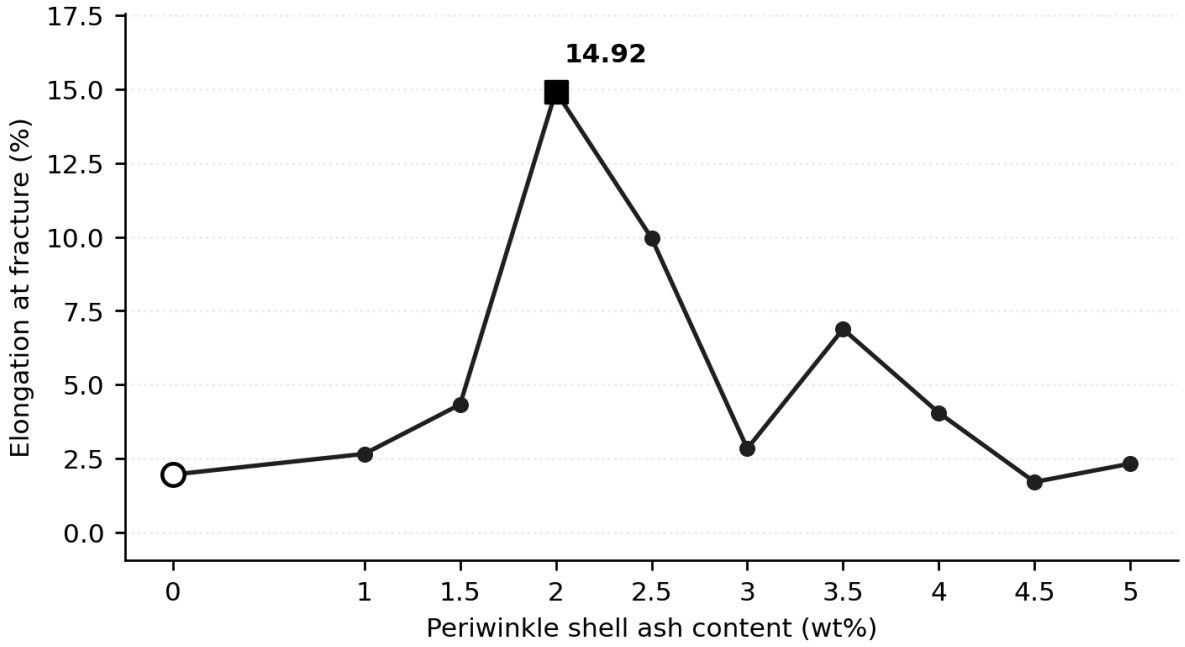

Figure 4. Percentage elongation against periwinkle shell ash content, transcribed from the project results chart.

Percentage elongation increased from 1.96% in the control to 2.66% at 1 wt% and 4.33% at 1.5 wt%. A much higher value of 14.92% was reported at 2 wt%, which is 12.96 percentage points higher or about 7.6 times the control value. At 2.5 wt%, elongation remained comparatively high at 9.96%, then fell to 2.84% at 3 wt% (Figure 4). The sequence shows that the composition most suitable for elongation in the chart available was 2 wt%, not the composition that gave the highest reinforced tensile strength. A higher elongation at failure does not necessarily mean that the material has a higher capacity for plastic deformation or fracture toughness. Huang et al. [3] pointed out that it is important to differentiate uniform deformation from fracture elongation in zinc-based materials. Here, no engineering stress–strain curve, gauge geometry or fracture assessment was available, so the large 2 wt% value should be considered a promising reported observation that needs to be confirmed, rather than evidence of an implant-grade ductility response.

4.5 Impact-test record and its unresolved scale

The original experimental programme included impact resistance, and the plotted series seems to peak at 2.5 wt% ash. The graph, however, shows 38.76 joules on an axis labelled “impact in joules” at that content and 32.50 joules at the control, while the project summary shows 5.18 joules at that content and 3.97 joules at the control. These pairs are not acceptable as raw measured energies without further information about the scale, specimen correction or units. This paper therefore does not attempt to determine percentages of impact improvement, rank compositions by toughness, or rely on the impact series to prove a reinforcement benefit. This outcome cannot be published quantitatively without the original test sheets or the printout from the impact machine.

4.6 Cross-property implications and external comparison

Table 2. Descriptive comparison of the most favourable reported values with the unreinforced alloy.

Measure	Control	Notable result	Change vs control
Comp.	75.73 MPa	83.92 MPa at 2%	+10.8%
Tensile	69.23 MPa	43.85 MPa at 4%	−36.7%
Elong.	1.96%	14.92% at 2%	+661.2%

The table clearly shows the performance trade-offs. Stresses of compression and elongation were the highest reported at 2 wt%, while the greatest tensile result was provided by the unreinforced control. Therefore, 2 wt% ash is considered good for the two reported parameters but not as an overall optimum value. To be able to select a service would necessitate weighted variations and predetermined strengths and deformations for other functional requirements. The same studies prove a modification of mechanical behaviour in the case of shell based reinforcement, but do not demonstrate the mechanism in the present samples. Umunakwe et al. [4] analyzed the aluminium composites and gave proof of the microstructures, while Olorunfemi et al. [6] examined the hybrid reinforcement of ash in another aluminium matrix. These results are comparators for the processing sensitivity, and not validation of zinc-alloy strengthening. Bala et al. [7] have studied corrosive exposure in a system prepared separately, and arrived at the conclusion that if corrosive degradation is an issue, mechanical performance cannot be used to determine suitability. The limits are more conservative when it comes to orthopedic relevance. The biodegradable orthopedic implant research was reviewed and it is reported that in-vitro and in-vivo degradation and biological response along with mechanical behaviour should be taken into consideration [8]. The present samples were not evaluated in terms of the amount of ion release, corrosion in simulated body fluid, cytocompatibility or long-term retained strength. In addition, the uncertain alloy composition prevents a trustworthy assessment of potentially undesirable alloying constituents. The work is not really a demonstration of an appropriate implant material, but rather a preliminary characterization of materials, perhaps with relevance to biomedical applications.

4.7 Limitations and minimum verification requirements

A few qualifications have to be made of the findings. First, although the report of fabrication mentions three rods for each composition, no spreadsheet showing the test level replicates, standard deviations or measurement uncertainties were provided. Confidence intervals cannot then be constructed for the apparent maximum and differences should not be described as being "statistically significant. Second, casting and test settings were not adequate for the source to allow for exact repeat and the very low compression of 3 wt% needs to be targeted for retest. Third, the impact-energy values are conflicting, making a reliable comparison between the impact response of compositions difficult. Machine output of each specimen should be preserved for a next series of experiments, specimen size and dimensions should be documented, the testing standards should be documented, casting temperatures, stirring and stirring speed should be documented and the particle distribution, porosity and fracture surfaces should be observed in the next series of experiments using a microscope. It would only be after these tests that replicate-based statistical tests could be applied or discusssion would be possible regarding strengthening and failure mechanisms responsible for mechanical fluctuations.

5 Conclusion

The experiment demonstrated that compressive, tensile and elongation properties of a cast alloy labelled as zinc-based were significantly different with periwinkle shell ash additions of 0-5 wt%. The maximum compressive stress recorded by chart was 83.92MPa at 2wt% whereas compressive stress recorded by chart for the Control was 75.73MPa. The elongation was also found to be the highest, 14.92%, at 2 wt%. There was no improvement in tensile strength with these observations, with the control reaching 69.23 MPa and the highest reinforced tensile strength achieved at 4 wt% being 43.85 MPa. This central result is thus not a uniform strengthening, but a system of composition-dependent trade-offs, combined with large fluctuations which are not explained by the reported measurements. The dataset can be used for a descriptive comparison and promising compositions can be found that can be verified further, but a statistically determined optimum composition cannot be determined, nor is a reinforcement mechanism guaranteed. Before the impact response can be compared quantitatively, the conflicting values of impact energy have to be resolved. Before any orthopedic applications are suggested, further work is warranted that involves reproducible casting, a repeat of mechanical tests, and microstructural examination and environmental testing.

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