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Inner architecture of bonded splats under combined high pressure and shear

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Abstract

The inner architecture of material plays a seminal role in its overall properties. Micron size commercial purity aluminium particles are deformed into an assembly of "interlocked splats" at room temperature under a combination of high pressure and shear. This results in fabrication of highly dense, strong and ductile samples. Dissections of the sample at different radii and planes are examined to construct a "lookout map" featuring *splat shaped* deformed particles to study the induced inner architecture. The structure comprises of splats of large "radial to axial aspect ratio", spread in parallel planes normal to the cylinder's axis. Micro shear punch (MSP) tests are carried out to characterize the heterogeneous strength properties and the radial distribution of material's density. A dimensional formulation is developed to interpret the MSP test data, taking into account some key parameters of the structure. The consolidated samples show an excellent shear strength; stronger than its solid counterpart and a pattern of radial strength distribution which is different with that of a similar solid sample.

Key words: Metal forming and shaping; powder technology; deformation and fracture; inner architecture

Introduction

Natural "Inner Architecture" (IA) in biological structures has inspired researchers to apply the natural design principles into man-made materials[1][2][3][4]. Particle-particle bonding, as the base for fabrication of many products directly from powder, has potentials to produce products with artificial inner architecture[5][6]. A possible scenario is mechanical bonding of particles resulted by their concurrent arranging, deforming and "grain refining"[7][8] to enhance strength and ductility of the product. The development of architectured materials often requires new processes (e.g. [9]).

High Temperature Superconductive (HTSC) tapes are produced from brittle "Bismuth Strontium Calcium Copper Oxide" using a "powder in tube" process [10]. Satisfactory performance of the tapes depends on grain alignment. Eliminating the heat treatment improves the "weak links" which is a major goal of researchers concerned with making HTSC tapes. A room temperature *particle bonding* without metallurgical interaction at the interfaces can be a new paradigm for material design. Eliminating heat treatment reduces carbon footprint and improves production rate for parts.

Combined shear and high pressures are commonly employed to produce bulk nano-structured materials (e.g. equal channel angular pressing with additional extrusion step[11] and a novel two step High Pressure Torsion (HPT)[12]). A review on this topic identifies the limitations on grain refinement for single-phase and multi-phase materials using severe plastic deformations[13]. Reducing the processing requirements (e.g. a high pressure and processing time) is desirable and makes the processes more practical.

We produced highly dense solid samples composed of *streamlined deformed particles*, hereafter called "*splats*", at the presence of shear and high pressure with a strong *particle bonding* at room temperature using a Confined High Pressure Torsion (CHPT) processing. It requires low-moderate processing (only 1.5 GPa pressure and 2 rotations), contrary to the conventional HPT processes which require processing under 2.5-15 GPa with 5-60 rotations [14][15][16][17][18][19]. Moreover this process minimizes the drawbacks in the conventional HPT process such as significant flash out of materials [20], effect of misalignment of anvils [20][21] and presence of dead zone [22][21].

To demonstrate the compliance of our processing with the proposed paradigm, some key parameters of its IA (excluding grain refinement in the particles) are discussed. These include particles' aspect ratio, compaction density, shear strength and their distribution. Effectiveness of the resulted IA is assessed by the arrangements of the processed particles and the excellent mechanical properties in the fabricated samples. The work also signifies the strength of the sample, as a function of chosen IA parameters. The optimum IA parameters have been studied analytically and experimentally.

A dimensional analysis is developed to understand the interaction of IA parameters in the sample.

Experimental results and discussion

Fabrication process and microstructure of the sample: **Fig. 1** shows SEM image of aluminium powder particles (99.7% purity) and basics of Confined High Pressure Torsion (CHPT). The as received Al powder is ECKA Granules® 75s with bulk density of 1250 kgm^{-3} and average particle diameter of 28.0 µm. The samples with $r_o = 8$mm and $t = 2$mm were produced, applying 1.5GPa pressure and two rotations (Fig. 1).

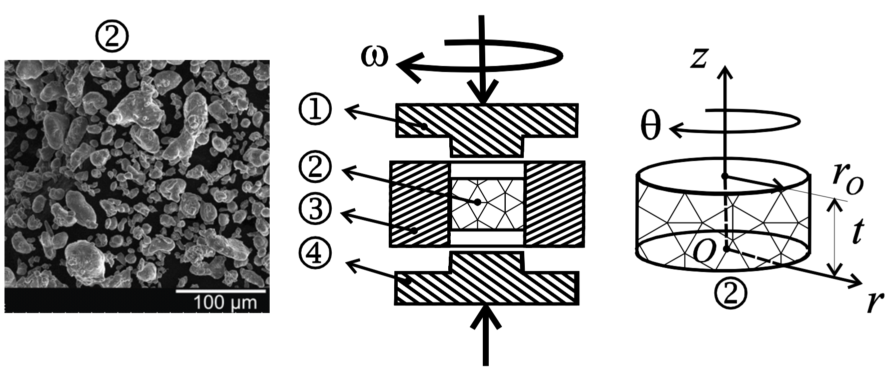

Fig. 1. Powder compaction using CHPT; left: SEM image of the Al particles before compaction, middle: 1 top punch (rotary), 2 CHPT sample, 3 constraining die and 4 lower punch (stationary), right: the coordinate system and dimensions.

A “lookout map”, a set of images showing the *splats’ boundaries* across different radii and orientations, is shown in **Fig 2**. It shows distribution of splats’ shape in the sample in two orthogonal planes along the radial and axial directions. Fig. 2-1 and 2 show the distribution, normal to z, along r axis. Elongated splats in Fig. 2-2 near the sample’s periphery have a large aspect ratio of $l_\theta/l_r \gg 1$ (l_θ and l_r are splat length in θ and r directions respectively). Figs. 2-3 to 2-5 show the distributions, normal to r, along r axis. Elongated splats in Fig. 6-5 near the periphery have a large aspect ratio of $l_\theta/l_z \gg 1$ ($l_z$is splat length in z direction). Inset of Fig. 2-6, shows boundaries for a splat and its inner substructure together. To explore a typical “triple-junction” at the interface of three splats, careful preparation of the interface is necessary to keep the void intact. A Focused Ion Beam (FIB) preparation and imaging was used here (Fig. 2-7) to minimize alteration of the gap/junction and the boundaries by mechanical/ chemical preparations.

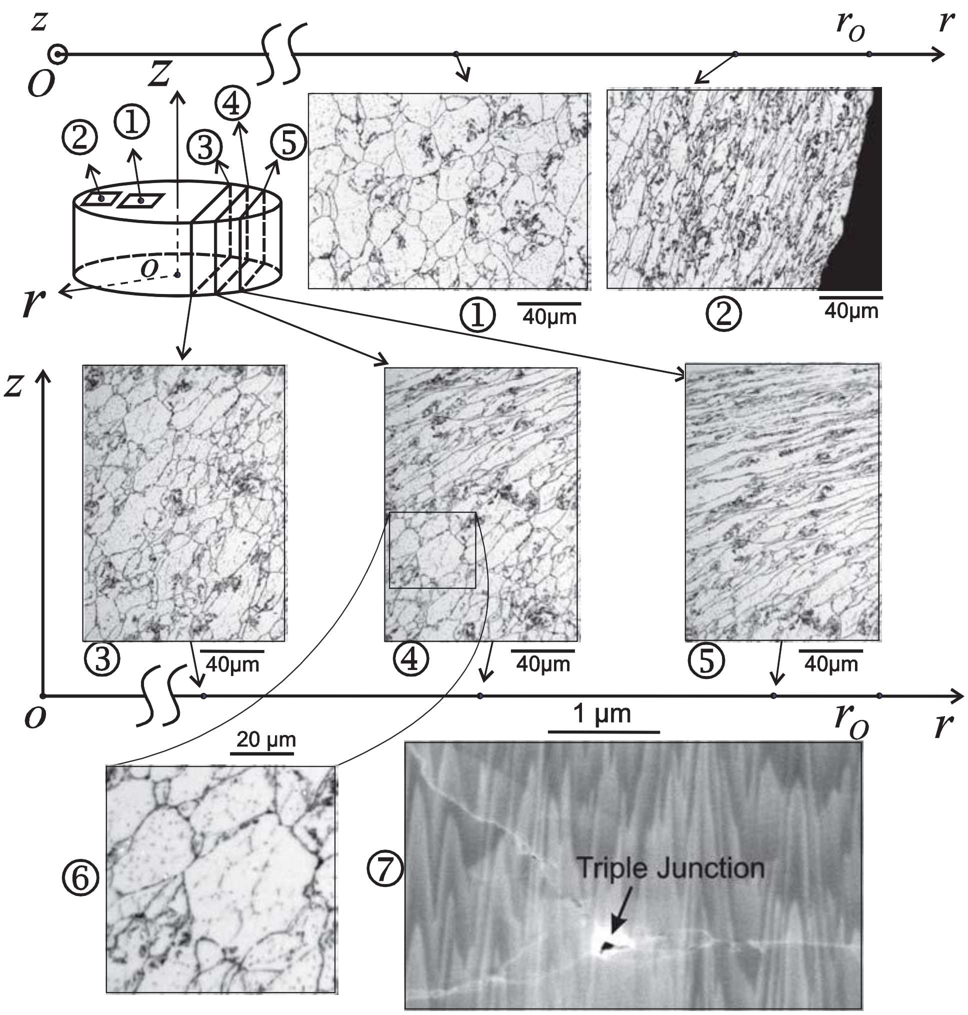

Fig. 2. The lookout map; Top left-location and orientation of the images in the sample.
1 and 2: optical micrographs near the centre and periphery, respectively (normal to z).
3, 4 and 5: optical micrographs near the centre (normal to r), middle and periphery of the sample, respectively.6: an inset of 4. 7: a FIB dissection of a triple-junction.

Both "*Splat boundaries"*(shown in Fig. 2 as dark solid lines)and "*grain structures"* are key to study the IA of the sample as they are responsible for an effective interlocking and splat's strength, respectively. Fig.2-6 also shows substructures inside the corresponding splats of Fig. 2-4. To explore the nature of such sub-structures and the extent of grain refinement is beyond the scope of current work.

The process results in spreading and elongating *particles* by deformation and interlocking them together. As shown in Fig. 2,the splats' "spreading and thinning" normal to z axisincreases by increasing their distance from the centre, r. Splats have random shape near the sample's centre and elongate near the edges with their aspect ratio of $l_\theta/l_z \gg 1$ and $l_r/l_z \gg 1$. As a result, a good mechanical interlocking can be expected. For a quantitative assessment of the property distribution in the radial direction, the shearing strength behaviour, micro-hardness and density at different radii are measured, analysed and explained next.

Characterizing distribution of in situ mechanical properties of the sample: For benchmarking of the sample's strength and its distribution in the radial direction, three *sample types* were used: 1. CHPT sample, 2. commercially pure Al (CP-Al) solid sample and 3. aluminum alloy Al5005-H34 solid sample. Tests are performed at different radii to investigate the radial distribution of the samples' shear strength, micro-hardness and density.

Micro shear punch (MSP)test: The fabricated samples are relatively small, the MSP test [23]is used here as a "small specimen test" technique[24] to assess the sample's shear strength at localized regions. **Fig. 3a** shows the MSP schematic and the positioning of the CHPT sample in the setup with five sampling points of different radii; r_1, r_2, … and r_5 (H1, H2, … and H5, respectively-$0 \leq r_i \leq 6mm$)with a punch radius of $r_p = 1.5mm$. Fig. 3b shows a SEM image of a blanked out MSP's piece. Typical "fracture" and "burnish" zones around the piece (shown as insets of 3c and d, respectively) are visible; the latter comprises almost 40% of the total 2mm width which indicates a ductile fracture. The MSP tests were also undertaken for the 2nd and 3rd sample types; for the Al 5005–H34 sample, the 3rd type, tests were carried out at two different point T1 and T2 (Fig. 3e) to confirm material's homogeneity and the test's repeatability. All of the tests were performed with a strain rate of $\dot{\varepsilon} = 0.01$ s^{-1}.The results, compared with those of the CHPT sample (type1), are shown in Fig. 3e.

Density: Densities of the MSPT's blanked pieces, ρ_i (i =1 to 5), were measured (Fig. 3f). Central part of the sample, $0 \leq r_i \leq$ 3.5mm, had the highest density ratio (~98%) which dropped sharply to

90% outside the zone ($3.5 \leq r_i \leq 6.0$ mm). Void collapse, as a main mechanism for the compaction, is easier in the central zone where the "radial rearrangement" is less restricted. A limited rearrangement near the edges, where the particles could only rearrange inward, explains its lower densification compared to that of the centre. Less porosity, increases density and decreases a non-hydrostatic pressure status in the media which is a pre-requisite for particle's super-plasticity. Very few *triple-junctions* of $\leq 0.1\mu m$ in size (Fig.2-7) indicate a high compaction (density ratio), a good mechanical interlocking and a high strength for the sample.

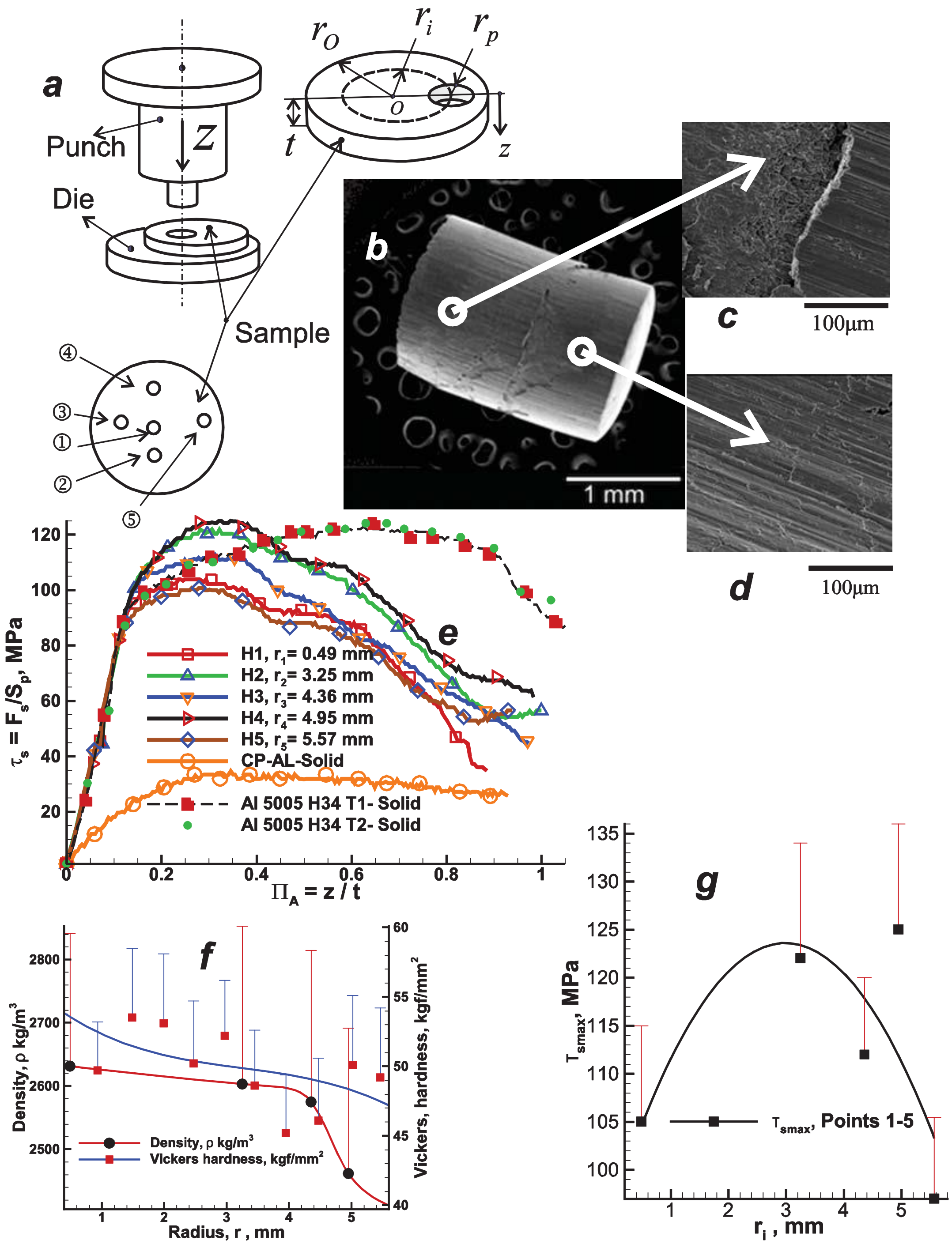

Fig. 3. a) Micro shear punch tests at five sampling points H1 to H5, b) a blanked piece, c) brittle fracture zone, d) ductile fracture zone, e) shearing strength at H1 to H5 for the 3 sample types, f) density and Vickers hardness in radial direction and g) maximum shearing stress vs. r_i at H1 to H5.

Micro hardness: Micro hardness across the samples' mid-planes along radial direction was measured using 10g load with 15 sec dwelling time using a Matsuzawa micro hardness tester MHT-1 according to ASTM: E384 11e1 to prevent the variation in hardness distribution in top and bottom surfaces .The results, shown in Fig. 3f, indicate that Vickers hardness drops moderately from its maximum ~53 kgf/mm^2 at the center to ~47 kgf/mm^2at the sample's edge. The average hardness of CP-Al and Al5005-H34 samples was also measured to compare with the processed sample. The pure CP-Al sample had an average hardness of 17.7 kgf/mm^2 which is significantly lower than that of the processed sample. Also, the Al5005-H34 sample had an average hardness of 53.2 kgf/mm^2 which is close to the highest hardness for the processed material (Fig. 3f).

A micro hardness measurement, with a small 10g load, is an indication of the surface strength instead of specimen's overall strength across its thickness. Care must be taken when interpreting such measurements due to the likely presence of pores in the vicinity of the intender. The measurement is also a point value which represents the surface strength immediately after the process. It is desirable to have an understanding of the overall mechanical properties of the sample across its thickness and to evaluate material's response under loading up to its failure. Given the small size of the sample, this was achieved here using the MSP test which are complementary to the hardness data.

For all sampling points H1-5, the CHPT sample's shearing strength, represented by the MSP response, is nearly "four times" higher than the solid CP-Al's strength and comparable with the Al 5005's strength. HPT processing of solid samples (e.g. [25]) produces a typical "radial gradient of strength". This reduces with an increase in the number of revolutions while for two revolutions, the gradient is significant. However according to Fig. 3e and 3 g, the CHPT sample's shearing strength shows a different gradient. The response is not homogeneous and changes in radial direction. In the specimen's central part, $0 \leq r_i \leq 5mm$, the shearing strength increases with radius (points 1 to 3 in Fig. 3g). A further increase of radius, points 4 and 5, reduces the maximum shearing strength. Despite some similarities between HPT and CHPT, their processed samples show different heterogeneity patterns.

The CHPT sample's IA is an open ended topic with an extensive list of parameters that impact the design. For example, the topic can be understood by studying the radial gradient of grain refinement in the splats. Also, a key question is the role of splat's aspect ratio and orientation on mechanical strength of the sample. The parameter sets considered here include: splat size, aspect ratio, density, MSP strength behaviour and hardness. To gather a preliminary understanding of the sample's performance, we develop a mathematical description for the MSP response taking into account the parameters measured at different radii. We utilize these, in the next section, in a dimensional analysis framework. Other aspects of IA are open for future works.

Similarity theory applied to the MSP response: Since the MSP response of the samples is governed by several parameters, a model is developed, using the similarity theory (e.g. [26]),to understand the interaction between them.

We define a parameter, λ_i, splat's representative aspect ratio at a sampling point i, as following:

$$\lambda_i = \frac{\bar{\lambda}_i}{\tilde{\lambda}} \tag{1}$$

Where $\bar{\lambda}_i$ is the average splat's aspect ratio in the punch's footprint and $\tilde{\lambda}$ is an optimum splat's aspect ratio for which the processed material has its maximum shearing strength. Also, we designate Vickers hardness at the punch's footprint for the i^{th} sampling point by H_i. It is assumed that MSP force, F_s, mainly depends on effective strain rate $\dot{\varepsilon}$, "density ρ_i at punch's footprint", "punch's instantaneous stroke (penetration)z", λ_i, H_i, "sample thickness t" and "the MSP's shearing area $S_p = 2\pi r_p t$":

$$F_s = F_s\left(\dot{\varepsilon}, \rho_i, z, t, S_p, H_i, \lambda_i\right) \tag{2}$$

Using "Buckingham-Π theorem", five dimensionless parameters, $\Pi_A = z/t$, $\Pi_B = \dot{\varepsilon}^2 t^2 \rho / H_i$, $\Pi_C = F_s/(\,H_i t^2)$,$\Pi_D = S_p/t^2$ and $\Pi_E = \lambda_i$are identified. A correlation function, f, is defined between the parameters as:

$$\frac{F_s}{H_i t^2} = f\left(\frac{z}{t}, \frac{\dot{\varepsilon}^2 t^2 \rho}{H_i}, \frac{S_p}{t^2}\ , \lambda_i\right) \tag{3}$$

To develop an expression for F_s as a function of radius r_i (Fig. 3e), we define a compound dimensionless parameter which is the product of four dimensionless parameters as following:

$$\Pi_B.\Pi_C.\Pi_D.\Pi_E = \frac{\dot{\varepsilon}^2 \lambda_i S_p \rho F_s}{{H_i}^2 t^2} \tag{4}$$

Two MSP tests at the two sampling points, r_1 and r_2 ($r_1 < r_2$), are considered here with identical values of$\dot{\varepsilon}$, S_p and t. It can be shown that two compound parameters corresponding to the points are equal. Consequently, a ratio of $F_s$for the two tests can be expressed as:

$$\frac{(F_s)_{r_1}}{(F_s)_{r_2}} = \left(\frac{H_1}{H_2}\right)^2 \times \frac{\lambda_2}{\lambda_1} \times \frac{\rho_2}{\rho_1} \qquad (r_1 < r_2) \tag{5}$$

A quadratic behaviour of$F_s$vs.$r$in Fig. 3g indicates that materials shearing strength is maximum at $r \approx 3.95$mm. Given the unimodal (descending) behaviours of H and ρ vs. r (Fig. 3e) and Eq. 5, it can be concluded that λ_i should change quadratically with radius, r. If $\tilde{\lambda}$is located at $r = \tilde{r}$, the conclusion may be restated as: for $0 < r < \tilde{r}$ an increase of λ_i increases materials' shearing strength and beyon this point ($\tilde{r} \leq r \leq r_o$) an increase of $\lambda_i$reduces the strength. Finding the exact location of $\tilde{r}$ is complex as $F_s$represents the response of a relatively large footprint.

The CHPT produces considerable friction induced shear between the particles. This fabricates highly heterogeneous and interlocked "splats" and a strong bulk sample. Equation 3 and its special cases (e.g. Eq. 4) provide insight on how mechanical properties of the sample are correlated. Splat's aspect ratio,λ_i, may be considered as a parameter related to the interlocking of the splats which has a pronounced effect on the material's shearing strength.

Further works are needed to explore other aspects of the IA such as "grain structure changes" during the process and their impact on the final strength.

Conclusions

Strong and dense samples were fabricated using micron size Al particles processed by the CHPT at room temperature under 1.5GPa pressure and two full revolutions. The splats' IA and their impact on the sample's properties were explored. Lookout maps showed streamlined splats, in planes normal to z axis and almost equiaxed in the sample's centre. Increasing their distance from the centre, splats were elongated and thinned with their aspect ratios of $l_\theta/l_z \gg 1$ and $l_r/l_z \gg 1$ near the edges. Formation of *very few triple-junctions* indicated a high densification with a maximum compaction ratio of98%near the centre of the sample. The CHPT sample revealed a ductile fracture during the MSP tests at different radii. Also sample's density and hardness decreased monotonically along the radial direction with their maximums at the centre and a sharp reduction of density near the periphery. Conducting MSP tests for solid samples of CP-Al and Al-5005-H34 showed that the CHPT sample was ~4 times stronger than the former, comparable with the later in shear and less formable than the both solid samples. Heterogeneous distribution and aspect ratios of splats contributed in their mechanical interlocking and the high strength of the CHPT sample evidenced by the MSP results. A similarity theory was employed to correlate the IA parameters of the sample and to interpret the strength results. It was concluded that there is a limit in improving shearing strength of the processed material by increasing the splat's aspect ratio. Increasing the aspect ratio beyond this limit, near the fabricated sample's periphery, reduces the sample's shearing strength.

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