

# SYNTHESIS & CHARACTERIZATION OF STAINLESS STEEL FOAM VIA POWDER METALLURGY TAKING CRUSHED UREA AS SPACE HOLDER

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## Abstract

Stainless steel foams are produced via powder metallurgy process taking crushed urea as space holder contributing porosity with 40-60 volume %. The pores formed by taking crushed urea as space holder produces nearly circular voids & they shrink after sintering. With increase in porosity regularity starts diminishing in the stainless steel foam samples. The foam samples obtained taking crushed urea particles don't show any plateau region during compression test & are ductile in nature. The yield strength of the foam decreases with increase in porosity. Therefore the stainless steel foams manufactured using crushed urea particles can be used in light weight high strength applications e.g. structures.

**Keywords:** Crushed; Porosity; Powder metallurgy; Stainless steel foams; voids.

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## 1. INTRODUCTION

In modern era, metal foams have emerged as a new class of cellular material consisting of a metal phase containing dispersion of pores. & has become an interesting area for researchers since they possess unique combination of engineering properties such as high stiffness, low density, better impact resistance, high energy absorption potential during compressive deformation, better erosion resistance & ease in giving complex shape [1-3]. Ashby et al (2000) reported that metal foams exhibits high stiffness to weight & strength to weight ratio, thus providing weight savings & cost reduction too. The potential applications of metal foams were provided as a function of their porosity by Banhart. They possess wide range of applications such as low weight sandwich panels, energy absorbing equipment, packing material, structural constituent in air and spacecraft [3-6], biomedical prosthesis [1-3], automotive, ship building & process control domains. Metal foams can be used as bumpers e.g. for suburban railcars, trams or cars as they are excellent energy absorbers due to their special deformation mechanism, the formation of deformation bands [5]. Metal foams display a characteristic compressive stress-strain curve which can be divided to three stages. During small strains (<1-2%) metal foam deforms elastically; than a plateau of deformation (up to 60-80% strain), where the stress is almost constant. Finally, as the deformation reaches the densification strain, thereafter the densified foam deform plastically [5]. Since long plateau region is found at constant compressive stress, foams are suitable for absorbing energy. Most metal foams comprises of aluminium, nickel, magnesium, lead, copper, titanium, steel and even gold as matrix [33].

A group of heat resistant and corrosion steels containing at least 10.5% chromium is referred as stainless steel. Stainless steel foams are metallic materials with various physical and chemical properties that can be used for several purposes. These foams exhibits high energy absorption, wide range of strength to elastic modulus ratios & huge ductility [7, 8]. The stainless steel foams have their main applications as sandwich panels, structural elements & beam cores where low weight and large specific rigidity is needed [32]. Steel foams are favourable materials for crash protection, building ships and packaging. The high energy-absorption properties of stainless steel foams & their lower cost makes them prospective competitors. They can be used in heat exchangers in the form of foam covered tubes and also as deflector plate for thermal and acoustic applications [7,8].

In general, two processes have been invariably used for metal foaming: (1) the liquid metal route where foaming is accomplished by direct foaming of melt with gas or some foaming agent [2, 9–12] and (2) the powder metallurgy route where foaming is affected by foaming a sintered compact [2]. Other routes include sintering of hollow spheres [13] and pressing the material around filler followed by sintering with subsequent filler dissolution or decomposition [14–16, 17, 18]. Recently, experiments have been carried out in microgravity conditions to see its effect on the stability of foams and obtaining highly porous structures [19–22].

In 1943, the first metal foam was invented, by Benjamin Sosnick at San Francisco California. Metal foams were first produced through powder metallurgy at the Fraunhofer Institute in Bremen (Germany) [ref]. Fraunhofer IFAM & Fraunhofer USA Centre-Delaware had conducted further development & application of metal foam technology [23,

24]. PM based process known as space holder method can be used to manufacture foamed metals. The space holder method starts with blending of metal powders along a suitable space holder material, after that hydraulic compaction of mixture (e.g. uniaxial pressing) is done that produces metal powder-space holder mixture compact referred as green compact. The green compact is then heat treated at a relatively low temperature to release the space holder, resulting in an unfired cellular foam metal structure. Finally, the compact is sintered at relatively high temperatures to provide structural integrity. This method permits a direct manufacturing of required shape foamed components having comparatively uniform cellular structure and a high level of porosity (60-80%). In powder metallurgy, space holder techniques are usual and offers a broad extent of structure and properties. There are number of methods of eliminating space holders e.g. by leaching, shaking, or by pyrolysis. Various space-holder materials are used such as carbamide (urea) & NaCl particle [25-29].

The compression and behaviors of metal foams have been widely investigated in the recent years. Wang et al.[30] also investigated the effects of cell sizes on the compressive strength and energy absorption. Nieh et al.[31] studied the effects of cell morphology on the compressive properties of foams. For characterizing the energy absorption behaviour of stainless steel foams the compressive properties, like the plateau stress and the densification strain must be determined as a function of the properties of the foam. Different from the solid materials, compressive deformation mode of the cell structured materials changes from uniform plastic deformation to localization.

The purpose of our research paper are: synthesis of stainless steel foam through PM route, compression testing of stainless steel foam to find the plateau stress using stress-strain diagram & to experimentally characterize green compact, pre-sintered stainless steel foam & sintered stainless steel foams by scanning electron microscope.

## 2. EXPERIMENTAL PROCEDURE

Stainless steel powder (Type 316-L45, density 7.85g/cm<sup>3</sup>) and urea particles (average size-2mm, density 1.32g/cm<sup>3</sup>) are used as the starting material. Stainless steel powder and urea are weighted on electronic weighing machine in a required weight proportion. To prepare the first samples of crushed urea particle 90 % ( 22.5 gram) of Stainless steel powder is mixed with 10 % (2.5 gram) of urea particles to obtain the porosity in the range of 40%. After weighing in the electronic weighing machine, powders are put in the crucible and mixed manually for about 15 minutes. The second sample 84 % (21 gram) of stainless steel powder is mixed with the 16% (4 gram) of urea particles to find the porosity of the range 50 volume pct. To prepare third sample 80 % (20 gram) of stainless steel powder are mixed with the 20% (5 gram) of urea particles to obtain the porosity of the range 60%.

The mixture is then compacted in a cylindrical die of 10 mm

diameter at a pressure of 250 MPa. The die is placed in the hydraulic compacting machine and is compacted for a time period of 30 seconds. This method produces cylindrical shaped green compact. Preheating of the precursor is done in a tubular furnace at the temperature 350<sup>o</sup>C, for a time period of two and half hour.

In the sintering process the foam samples are heated up to 1100<sup>o</sup>C for a time period of 110 minute at a heating rate of 10<sup>o</sup>C/min. Then the samples are kept in the horizontal high temperature vacuum furnace for a time period of 60 min., so that whole parts of the samples are attend a constant temperature of 1100 °C. Then the samples are cooled in the furnace. Then the samples are kept in the furnace for a Time period of 60 min., so that whole parts of the samples are attend a constant temperature furnace.

The microstructure characterization is carried out on a JEOL 5600 Scanning Electron Microscope. The stainless steel foams mechanical properties are determined by executing compression test on the sample. At the strain rate of 10<sup>-3</sup> s<sup>-1</sup>, the compression test is performed on Instron universal testing machine

## 3. RESULT AND DISCUSSION

During cold compaction, green compacts measurements are taken with the help of the vernier caliper and weighed by the electronic weighing machine. The theoretical density of the powder mixture is calculated using the mixture rule formula.

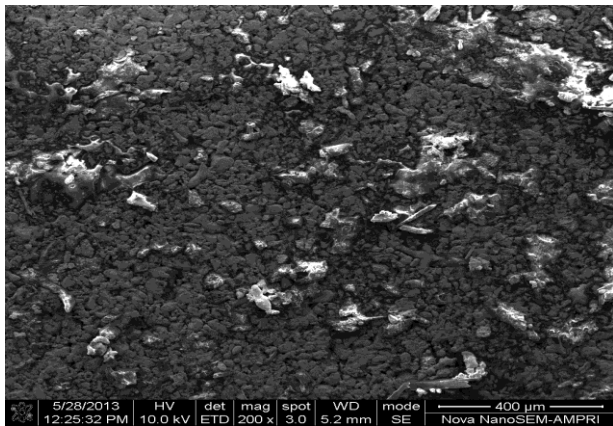
$$\frac{1}{\text{Theoretical Density}} = \frac{\text{wt\% of ss}}{\text{density of ss}} + \frac{\text{wt\% of urea}}{\text{density of urea}}$$

$$\text{Green density} = \frac{\text{wt of mixture}}{\text{volume of cold compact}}$$

The green compact is observed on the compacted surface using FE-SEM. The micrograph shows the dispersal of urea particles in the stainless steel matrix. In the image the black regions are the stainless steel particles while the white regions are the urea particles embedded in the matrix.

**Table 1** showing various measurement data of stainless steel green compacts mixed with crushed urea particles

Sample no.	ss (wt %)	Urea (wt %)	Weight of mixture (gm)	Volume of cold compact (cm <sup>3</sup> )	Green Density (gm/cm <sup>3</sup> )	Theoretical Density (gm/cm <sup>3</sup> )	Green Density (%Theoretical Density)
1	90	10	4.7	1.096	4.288	5.251	81.66
2	90	10	4.08	0.98	4.163	5.251	79.28
3	90	10	4.15	1.041	3.984	5.251	75.87
4	84	16	3.69	0.948	3.891	4.381	88.81
5	84	16	3.73	0.969	3.847	4.381	87.81
6	84	16	3.77	0.949	3.969	4.381	90.59
7	80	20	3.68	0.945	3.618	3.946	91.68
8	80	20	3.75	0.985	3.806	3.946	96.45
9	80	20	3.4	0.93	3.655	3.946	92.62

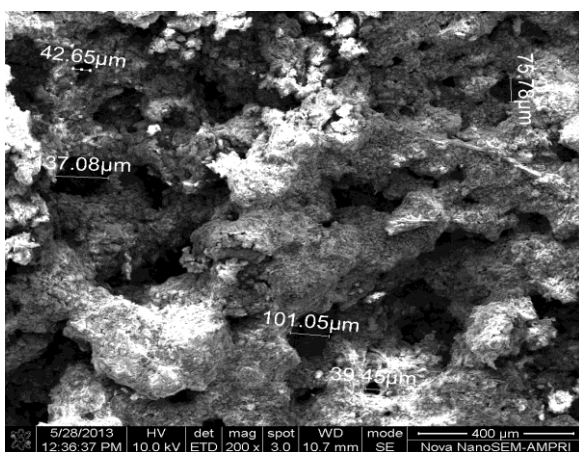


**Fig 1** showing SEM image of crushed urea particle embedded in the stainless steel matrix

During preheating, the green compact is placed in the furnace on the ceramic plate. After the temperature reached 370 °C, the green compacts are soaked for 2.5 hrs and then cooled in the furnace. Due to the evaporation of the urea, from the compacted part, pores are obtained.

**Table 2** showing various measurement data of stainless steel pre-heated compacts mixed with crushed urea particles

Sample no	Initial wt.(gm)	Final wt.(gm)	Volume of preheated compact(cm <sup>3</sup> )	Density of preheated compact(g m/cm <sup>3</sup> )	% Density	% porosity
1	4.7	4.27	0.889	4.8	61.2	38.83
2	4.08	3.41	0.731	4.66	59.4	40.56
3	4.15	3.55	0.753	4.71	60.1	39.9
4	3.69	2.51	0.625	4.01	51.1	48.89
5	3.73	2.57	0.655	3.92	50.1	49.95
6	3.77	3.03	0.778	3.89	49.6	50.4
7	3.42	2.75	0.802	3.42	43.6	56.44
8	3.75	2.94	0.869	3.38	43.1	56.95
9	3.4	2.72	0.871	3.12	39.8	60.21



**Fig 2** showing SEM image of preheated sample (crushed urea)

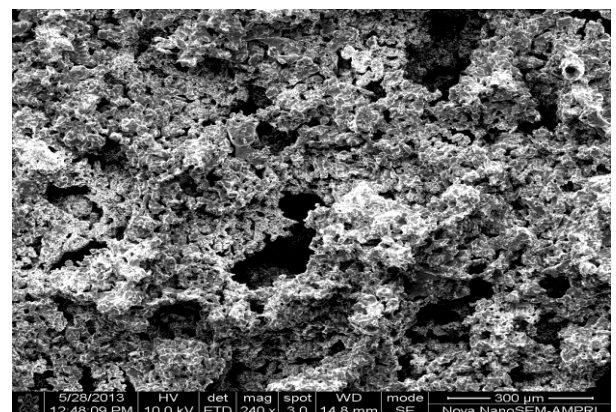
urea space holder)

The pores obtained are larger than that of urea particles used due to strain produced by heating and evaporation. The microstructure shows the distribution of pores in the stainless steel matrix. Microstructure shows after the removal of urea in the form of gas from compact as circular pores. Such pores are formed due to low pressure used during the compaction of the powder mixture & shape of urea particles is also a key factor. Pore size is larger than that of the size of the urea particles because urea particles evaporate after breaking the bonds with the stainless steel matrix resulting larger size voids. Sintering is done after preheating to give the strength and to develop metallurgical bonding within the steel matrix. The preheated part is heated below its melting point but higher than the preheating temperature. Due to high temperature heating, bonding between the stainless steel particles takes place which gives strength to the preheated compact.

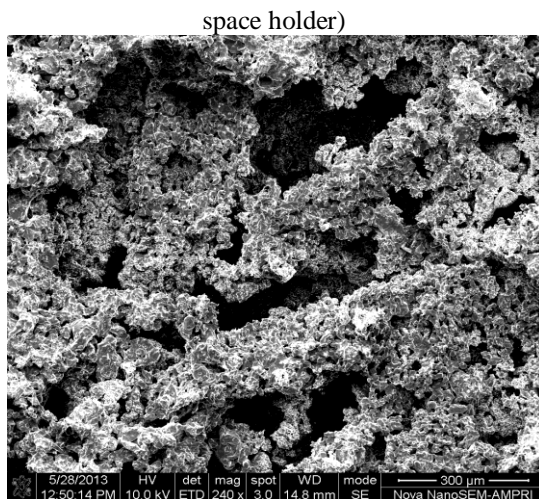
**Table 3** showing various measurement data of sintered foam samples (crushed urea space holder)

sample no	% S.S.	% Urea	Weight (gm)	Dia. (mm)	Height (mm)	Density (gm/cm <sup>3</sup> )	% Density	% porosity
1	90	10	3.86	9.8	10.7	4.788	61	39
2	90	10	4.22	9.8	12.1	4.6	58.6	41.4
3	90	10	3.35	9.8	9.67	4.631	59	41
4	84	16	2.51	9.7	9.37	3.611	46	54
5	84	16	2.43	9.8	8.64	3.768	48	52
6	84	16	2.96	9.7	10.3	3.885	49.5	50.5
7	80	20	2.89	9.6	11.3	3.528	44.94	55.1
8	80	20	2.87	9.6	11.6	3.441	43.83	56.2

After the sintering process is complete, the samples attained higher strength. Due to sintering process, the pore walls strengthens and the porosity is obtained in the samples. During the process of sintering the volume of the sample will shrink, although sometime it will expand due to the existence of internal stress produced in the compaction. The pores produced after sintering are smaller than that of the pores obtained after preheating. The greater driving force during sintering causes the pores to shrink to smaller size.

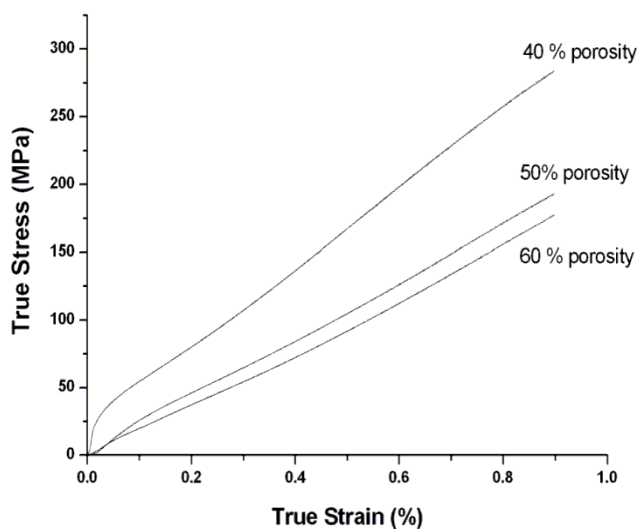


**Fig 3** showing SEM image of sintered sample (crushed urea)



**Fig 4** showing SEM image of sintered sample (crushed urea space holder)

The pores produced are circular in shape because crushed urea is used as space holder. Being smaller in size, the pores formed are circular in shape. Some pores are bigger and some pores are smaller it is due to uneven size of the crushed urea particles.



**Fig 4** showing True stress and True strain curve (crushed urea sample)

The compressive behavior of cellular metals is very interesting due to their high energy absorption capacity. The pores in this sample are nearly circular in shape. The plateau stress is not much distinct due to greater degree of uniform distribution of pores, larger young modulus & fcc structure (ductility) of austenitic stainless steel. & the yield strength decreases with increase in porosity due to decreasing material content & decrease in ductility.

## 5. FUTURE SCOPE

- By varying the compaction pressure we can observe the changes in microstructure and mechanical properties of the resulting foams with different porosity. Resulting applications should also be

discussed.

- The changes in true stress -strain diagram by varying the strain rate can also prove to be beneficial.
- While using different shaped space holder, micro structural changes could be observed.
- The sintering atmosphere variation may also prove to be an asset resulting in different bond strength between steel particles.
- Sintering temperature change can also produce better pore morphology in steel foams.
- Change in foam sample shape by using different space holder could also be done to produce better results.

## 6. CONCLUSION

- Stainless steel foams can be fruitfully produced through powder metallurgy using urea as space holder.
- The porosity level can be varied with the amount of space holder used.
- The sintering should be done in vacuum to prevent the oxide formation during sintering.
- The pore morphology depends on the space holder used, sintering temperature and sintering time.
- The stress-strain curves of compression of stainless steel foam with crushed urea particles as space holder doesn't show distinct plateau region.
- The processed stainless steel foams can be utilized in high strength structural applications.

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