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14. ABSTRACT It was the objective of this activity to make metal steel foam panels with low carbon steel face sheets by hot isostatic pressing. Panels were successfully made with strontium carbonate foaming agent by both HIP plus foam and HIP plus forge plus foam. Resulting foam density was 50%, representing the best steel foam to date using Fraunhofer USA Delaware technology. It is planned to apply foam steel structural panels in applications such as Naval vessel doors and Carrier elevators, where light weight and high specific stiffness are important.					
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**Phase I Final Report - N00014-99-M-0022 Office of Naval Research
Ultra-Light Structural Steel From Metal Foams**

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May 29, 1999

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Phase I Final Report - N00014-99-M-0022
Ultra-Light Structural Steel From Metal Foams

Outstanding results have been obtained in Phase I due to the excellent teamwork between UltraClad Corporation and Fraunhofer USA, Center for Manufacturing and Advanced Materials Delaware.

Abstract

Steel foams have also been produced on a laboratory scale; methods for scale-up to low cost manufacturing/fabrication via hot isostatic pressing have been investigated and demonstrated in this Phase I effort. UltraClad has made use of the Fraunhofer Institute (Delaware) foam technology via subcontract coupled with its own expertise in hot isostatic pressing, metal matrix composite technology(4,5), and scale-up of laboratory concepts to further develop steel cored foam panels. An application for a Phase II has been identified, a foam core steel door for a naval vessel constructed by Bath Iron Works.

Highlights of Phase I results include: Foam via HIP plus foaming and HIP plus forging plus foaming yielded the best steel foam density and mechanical property results to date in comparison with other steel foam processing routes. Identified problems to solve include: Understanding and control of less than ideal pore size distribution.

Introduction

Cellular materials are found in everyday uses for their high stiffness, low specific weight and other properties. Applications range from light-weight construction and packaging, to thermal insulation, vibration damping, and chemical filtration. Metallic cellular materials, namely metal foams, merit the use of cellular materials and are becoming a new class of engineering materials. These foams enlarge the application range of cellular materials because of their excellent physical and mechanical properties, as well as their relative amenability to recycling compared to polymeric foams.

A powder method for creating aluminum and other metal foams was invented at the Fraunhofer Institute for Applied Materials Research (IFAM) in Bremen, Germany, and improved upon by its US facility, the Fraunhofer Resource Center - Delaware (FRC-DE), especially for steel foaming(1,2). This method allows for low cost and direct net-shaped fabrication of foamed parts with a relatively homogeneous pore structure. Using a powder metallurgical method, it is now possible to obtain metallic foams of various metals and alloys with a complex geometry. Sandwich structures composed of a porous metallic aluminum foam core and metallic face sheets can also be produced. Mechanical and physical properties of foams have been studied and results look promising for many applications(3).

Steel foams have also been produced on a laboratory scale; methods for scale-up to low cost manufacturing/fabrication via hot isostatic pressing have been investigated in

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this Phase I effort. UltraClad has made use of the Fraunhofer foam technology via subcontract coupled with its own expertise in hot isostatic pressing, metal matrix composite technology(4,5), and scale-up of laboratory concepts to further develop steel cored foam panels.

Background

Metallic foams fabricated by this approach exhibit a closed-cell microstructure with higher mechanical strength than open-cell foams. This type of microstructure is particularly attractive for applications requiring reduced weight and energy absorption capabilities. Fraunhofer's prior experience in the production of foamed aluminum indicates that the foamed component provides a substantial increase in the stiffness/weight ratio (SWR) with a fractional density of as low as 20% of fully dense aluminum. The foam features a relatively homogeneous closed-cell microstructure

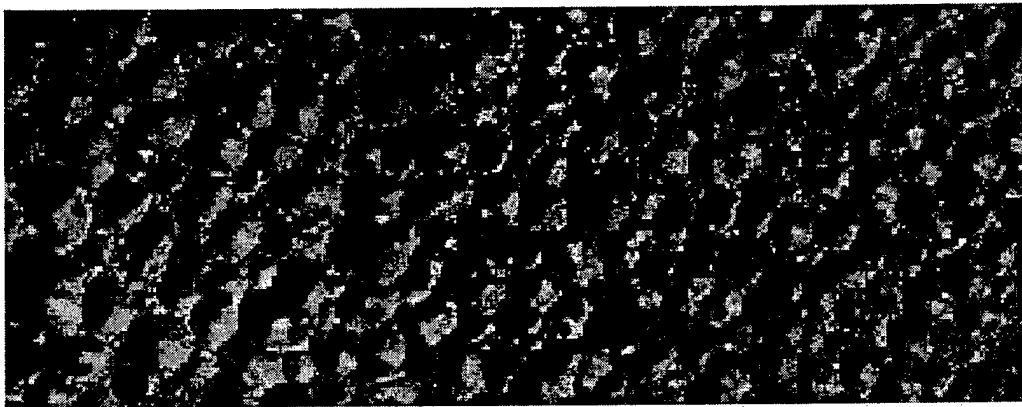


Figure 1: Typical aluminum foam microstructure

which is responsible for the high SWR of the foam. The many microscopic "I-beams" surrounding the pores provide the strength and high stiffness of the foamed structure (Figure 1). Under deformation this microstructure features a localized cell collapse and rapid strain energy dissipation which leads to unique deformation behaviors and material properties including high SWR and energy absorption in the material.

Metal foams have potential applications in the field of crush energy absorption used for land-based transportation, e.g. filling material for bumpers, strengthening and energy absorption material for A- and B-pillars, and fire walls in automobiles. A prototype concept vehicle with aluminum foam sandwiches as crush energy absorbers was demonstrated by Wilhelm Karmann, GmbH at the 1998 International Auto Show in Detroit, MI. Figure 3 shows the concept car presented at the International Auto Show. The panels for the front and back passenger compartments were made of aluminum foam sandwiches.

Advantages of Steel Foams: Compared to the aluminum foam sandwich structures used in automotive applications, steel foam sandwiches lead to an

improvement in the following properties:

- increased mechanical strength;
- increased specific stiffness;
- reduced thermal conductivity;
- increased melting point;
- and improved corrosion resistance.

In particular, the increased mechanical strength and melting point and improved corrosion resistance of the prospective steel foam sandwich structures are of interest for naval applications such as light-weight deck construction and doors. Steel foam sandwiches as light-weight material for landing decks or elevators have the potential to reduce weight and to provide the necessary stability against fire and corrosion.

Task 1: Identification and Prototyping of Steel Foam Systems and Panels
Carbon Steel Metal foaming experimental results. Work during this period has concentrated on foaming experiments with carbon steel and CrN foaming agents using the foaming matrix previously chosen and reprinted below in Table 1.

FoamingAgent	CompactDensity ~80%	CompactDensity ~80%	CompactDensity ~88%	CompactDensity ~88%
0.5 w.% CrN	HeatingRate 40°C/min	HeatingRate 60°C/min	HeatingRate 40°C/min	HeatingRate 60°C/min
1.0 w.% CrN	HeatingRate 40°C/min	HeatingRate 60°C/min	HeatingRate 40°C/min	HeatingRate 60°C/min

The following figures show the experimental results of the steel foaming experiments with CrN (chromium nitride) as the foaming agent. Anchor Steel 1000C powder was blended with CrN powder in a turbula blender, cold pressed in a steel die, and foamed and 1300°C for 4 minutes.

Experimental results and cross sectional photographs.

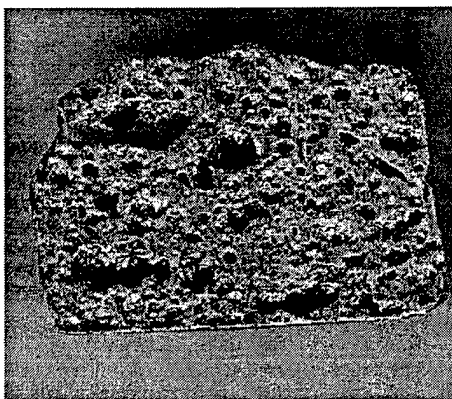


Figure 2: Steel Foam Sample 1
 0.5%CrN, Compact Density 84%
 Heating Rate 40°C/min
 Foam Fractional Density: 59%

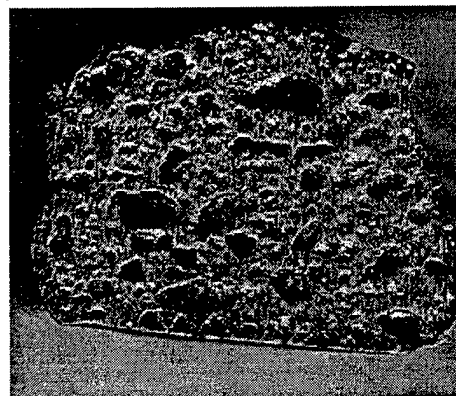


Figure 3: Steel Foam Sample 2
 0.5%CrN, Compact Density 88%
 Heating Rate 40°C/min
 Foam Fractional Density: 58%

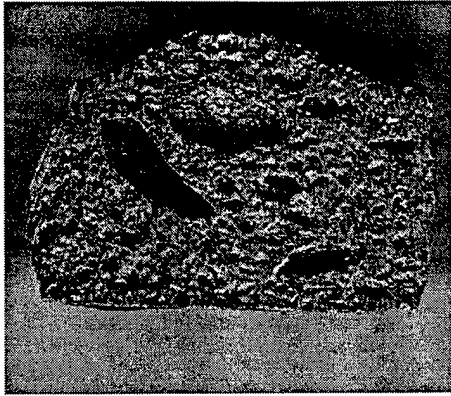


Figure 4: Steel Foam Sample 3
1.0%CrN, Compact Density 83%
Heating Rate 40°C/min
Foam Fractional Density: 63%

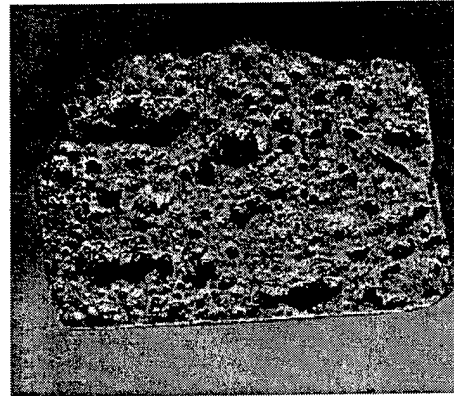


Figure 5: Steel Foam Sample 4
1.0%CrN, Compact Density 87%
Heating Rate 40°C/min
Foam Fractional Density: 65%

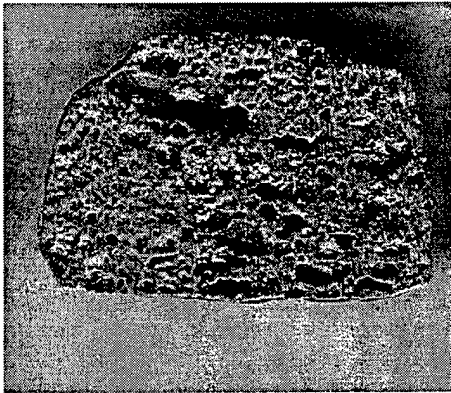


Figure 6: Steel Foam Sample 5
0.5%CrN, Compact Density 83%
Heating Rate 60°C/min
Foam Fractional Density: 60%

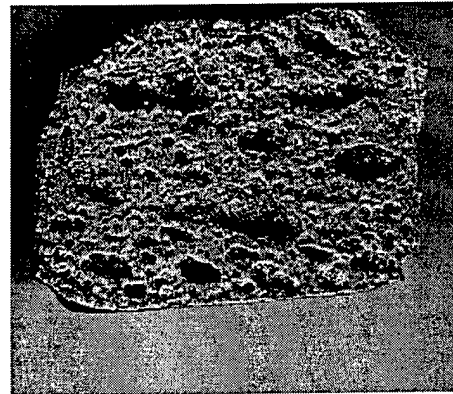


Figure 7: Steel Foam Sample 6
0.5%CrN, Compact Density 98%
Heating Rate 60°C/min
Foam Fractional Density: 58%

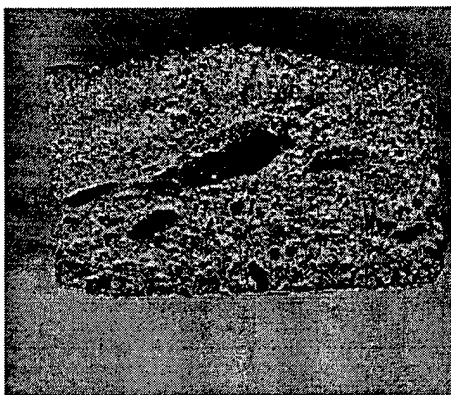


Figure 8: Steel Foam Sample 7;
1.0%CrN, Compact Density 82%
Heating Rate 60°C/min
Foam Fractional Density: 64%

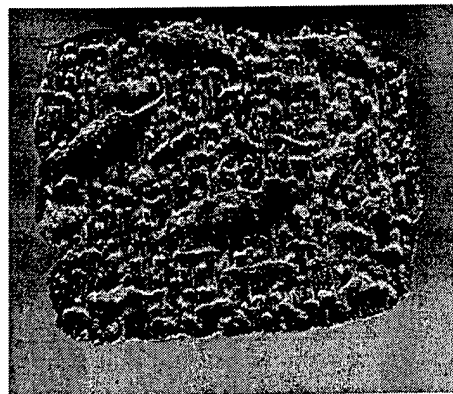


Figure 9: Steel Foam Sample 8;
1.0%CrN, Compact Density 92%
Heating Rate 60°C/min
Foam Fractional Density: 60%

Foaming Agent	Compact Density ~80%	Compact Density ~80%	Compact Density ~88%	Compact Density ~88%
0.5 w.% CrN	HeatingRate 40°C/min	HeatingRate 60°C/min	HeatingRate 40°C/min	HeatingRate 60°C/min
Foamed Density	(1) 59%	(5) 60%	(2) 58%	(6) 58%
1.0 w.% CrN	HeatingRate 40°C/min	HeatingRate 60°C/min	HeatingRate 40°C/min	HeatingRate 60°C/min
Foamed Density	(3) 63%	(7) 64%	(4) 64%	(8) 60%

Table 2. Tabulated summary of results for carbon steel CrN metal Foams

The following observations may be made from the test results:

- Carbon steel with 2.5 w.% carbon can be foamed with CrN as the foaming agent.
- To date, the resulting foam fractional densities range from 58%-64%.
- Based on subjective appearance of the pore uniformity and size distribution, It appears that the steel compacts with 0.5%CrN foam better than those with 1%CrN.
- Figure 2, which exhibited the lowest porosity, 58%, exhibited the best pore structure obtained in this series of experiments. It was also noticed that there are different sizes of pores in the foam structure. It was speculated that CrN might not be uniformly distributed within the compact after blending. The CrN particle distribution after blending and compaction will be evaluated using SEM-EDX mapping technique.

Low Carbon Steel Metal Foams

Additional recent testing focused on the compaction and foaming of carbon steel samples with lower carbon than 2.5%. Compaction of the powder mixtures was conducted in a mold made of grade H13 tool steel, and the pressing temperature was 550°C. The compacted samples had an average density of 7.25 g/cm³ (fractional density of 94%). The experimental parameters included the type and the amount of foaming agents. Three types of foaming agents were tested including SrCO₃, CrN, and Mo₂N. Figures 1-3 show the Differential Thermal Analysis (DTA) of each foaming

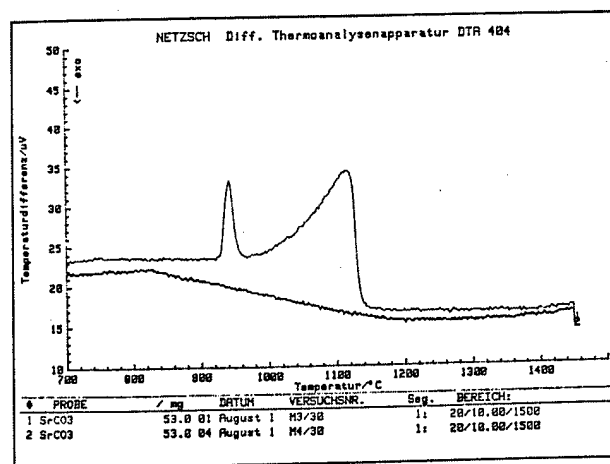


Figure 10: Differential Thermal Analysis of SrCO₃

agent. It can be seen that SrCO_3 begins to decompose at 910°C while the CrN begins at 1000°C and Mo_2N at 1300°C , respectively.

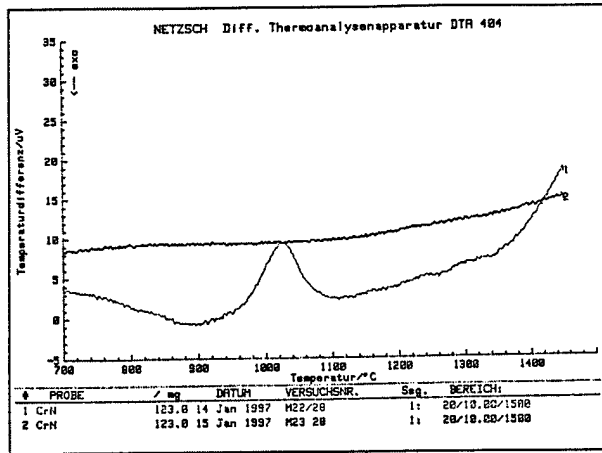


Figure 11: Differential Thermal Analysis of CrN

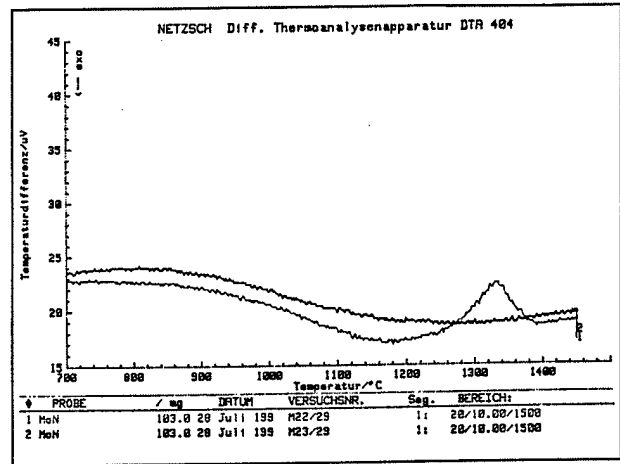


Figure 12: Differential Thermal Analysis of Mo_2N

A total of fourteen samples with 1 wt.% carbon were compacted and heated at three furnace set temperatures: 1400°C , 1425°C and 1450°C . The furnace temperature is limited to 1450°C under Ar due to insufficient power input to the furnace. In most of the experiments, samples did not melt or only partially melted on the sample surface. No melting in the samples was observed at the set-temperatures of 1400°C and 1425°C .

The following is a table listing the test samples, resultant densities and observations:

% Foaming Agent	<u>Compact Density</u> Final Density	Comment
0.5 w.% SrCO ₃	7.31 (95%) 6.93 (90%)	Heated in Ar at 40°C/min; no melting
0.5 w.% SrCO ₃	7.25 (94%) 6.58 (85%)	Heated in Ar at 60°C/min; no melting
1.0 w.% SrCO ₃	7.16 (93%) 5.67 (74%)	Heated in Ar at 40°C/min; partially melted
0.5 w.% CrN	7.19 (93%) 6.95 (90%)	Heated in Ar at 40°C/min; no melting
1.0 w.% Mo ₂ N	7.32 (95%) 6.46 (84%)	Heated in Ar at 40°C/min; no melting
0.5 w.% CrN	7.40 (96%) 6.40 (83%)	Heated in N ₂ at 40°C/min; partially melted
1.0 w.% Mo ₂ N	7.34 (95%) 6.11 (80%)	Heated in N ₂ at 40°C/min; partially melted

Table 3: Foaming experiments on 1.0% carbon steel conducted at 1450°C

Summary of results on foaming of 1% carbon Steels:

- It was observed that SrCO₃ decomposed and generated gas before furnace reached the set temperature of 1450°C. The generated gas leaked out without foaming the compact. Samples with CrN and Mo₂N did not show significant gas-leaking during heating.
- The heating capacity of the furnace used is limited to 1450°C under Ar and N₂. Future task will focus on lowering the melting temperatures of the carbon steel compacts by either increasing the carbon content (to 1.5 wt.%) or adding other alloying elements such as phosphorus to the mixture.

Results of Hot Pressing Experiments on Stainless Steel Powders

316L and 420 stainless steel powders were not successfully compacted in the 1.25" diameter mold. The powder mixture was compacted to less than 80% fractional

density and broke into several pieces. The compaction was also conducted at 550°C due to the temperature limitation of H13 steel mold. We suspect that these spherical stainless steel powders may require higher compaction pressure and broader powder size/distribution to achieve a high compaction density. Further experiments on stainless steel powders were performed by HIPing as described below.

Hot Isostatic Pressing of Carbon Steel Powder Panels with Foaming Agents.

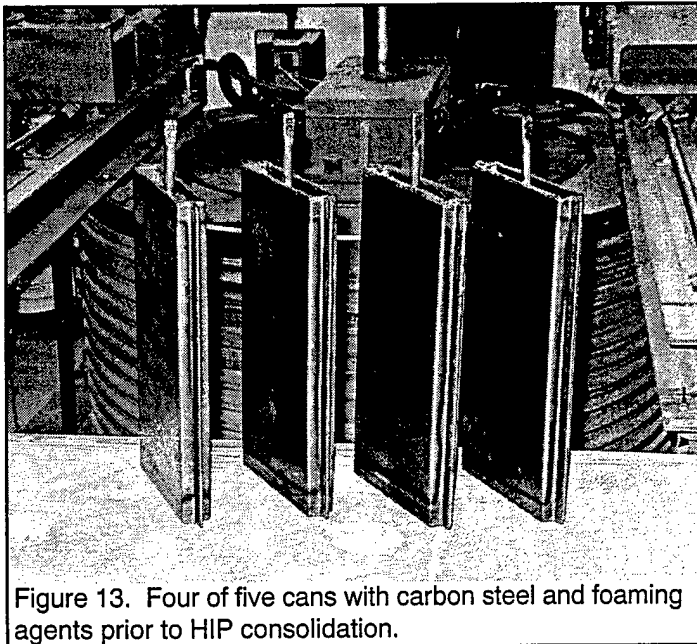


Figure 13. Four of five cans with carbon steel and foaming agents prior to HIP consolidation.

Five samples were prepared from carbon steel powders and foaming agents for compaction by hot (warm) isostatic pressing. Approximately 6 inch by 10 inch by 1 inch thick cans were prepared from 18 gauge 1018 low carbon steel by bending on a press brake and TIG welding, and filled with the powder foaming agent mixtures. The powders were blended in a turbula T10 blender for 1/2 hour each (tap density = 0.133lbs/in³). After filling the cans were evacuated and leak checked using a mass spectrometer helium

leak detector and then sealed by hot crimping.

The cans were consolidated at 705°C/2000 Bar/1 hour in a 16 inch diameter hot isostatic press. All cans consolidated properly, as shown in figures 15 and 16. Some evidence of lack of fill was noted on the ends of several cans, but there was no effect on resulting usability. The can dimensions after HIP suggested a consolidated density of 90-95%. This compares with a hot pressed density of 80-88% at Fraunhofer.

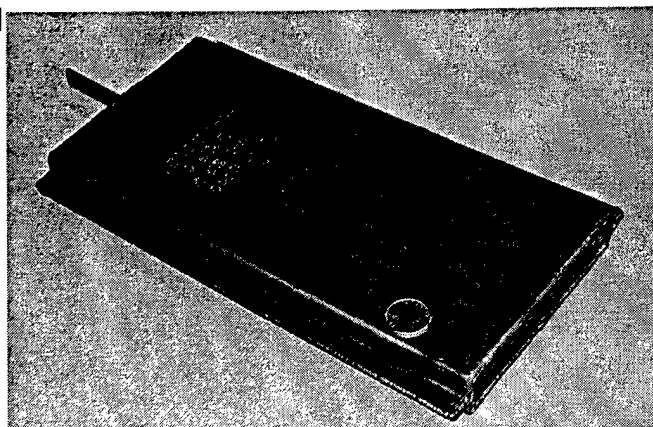


Figure 14. Can before consolidation.

Foaming Agent 1000C, %C	0.5 w.% CrN	1.0w%Mo ₂ N	0.5w.% CrN + 0.25w.% SrCO ₃
2.5 w.% C	X	X	X
1.0 w.% C	X	X	

Table 4. HIP Cabon Steel Experimental Matrix.

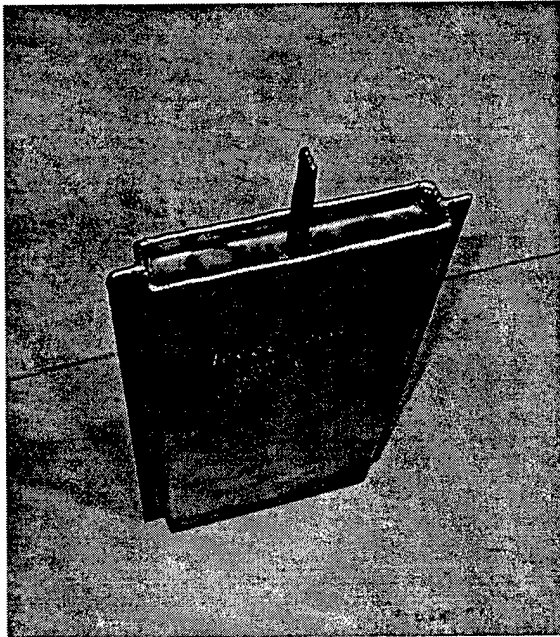


Figure 15. Can before HIP consolidation.

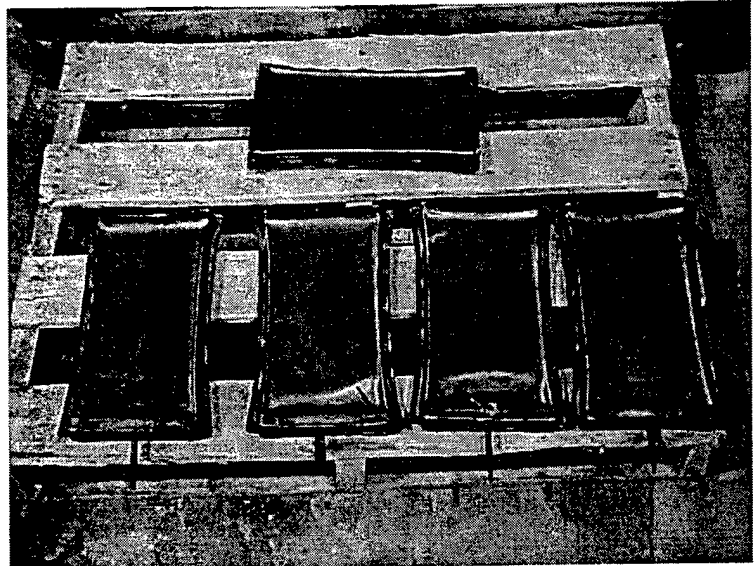


Figure 16. Five cans after consolidation at 705°C/2000Bar/1 hr.

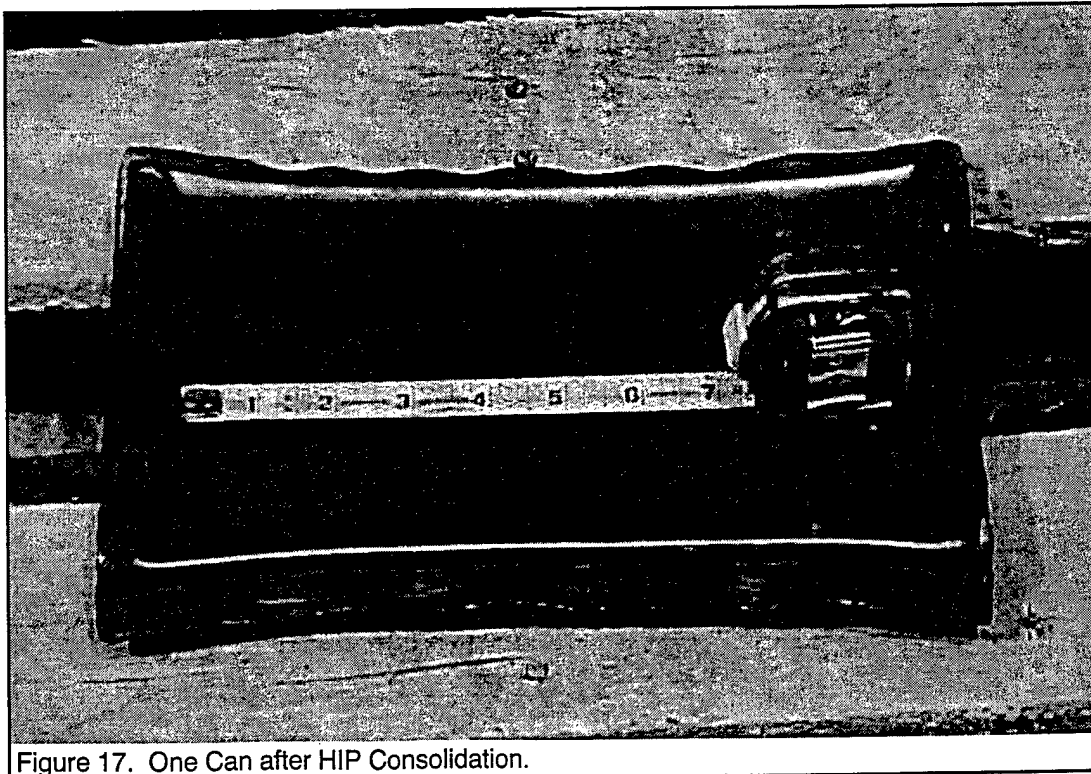


Figure 17. One Can after HIP Consolidation.

Results of Foaming of HIP Carbon steel samples.

Results of foaming of the HIP carbon steel samples were very positive. They represent the best foaming results to date on steel foams. The results of density are shown below in the table below:

Sample	Carbon, wt%	Foaming Agents, wt%	Foamed Results
1	2.5	0.25% SrCO ₃	Good ~ 50% density
2	2.5	0.5% CrN	Slight
3	2.5	0.25% SrCO ₃ + 0.5% CrN	Slight
4	1.0	0.5% CrN	None
5	1.0	1.0% Mo ₂ N	None

Table 5. Foaming results of HIP Carbon Steel Foam Panel Samples

Figure 18 and 19 show, respectively, a foam sandwich from sample 1 (HIP iron powder 0.25% SrCO₃ + 0.5% CrN) and a cross section of such a sandwich. Consistent fractional densities of 50% were obtained with samples from this condition. Examination of figure 19 shows good bonding between the steel foam core and the low carbon steel face sheets.

The samples #3 (HIP iron powder, 0.25% SrCO₃ + 0.5% CrN) were only slightly foamed. It was observed that the low carbon face sheets were slightly separated from the foamable iron during heating. This separation may have inhibited foaming by resulting in a nonuniform temperature distribution or another mechanism; more investigation is necessary. Similar phenomenon were observed for samples #2 with CrN as the foaming agent. Previous foaming experiments with hot pressed samples indicated that these compositions should foam successfully. Also no foaming was observed with HIP iron powder with 1% carbon and nitride foaming agents.

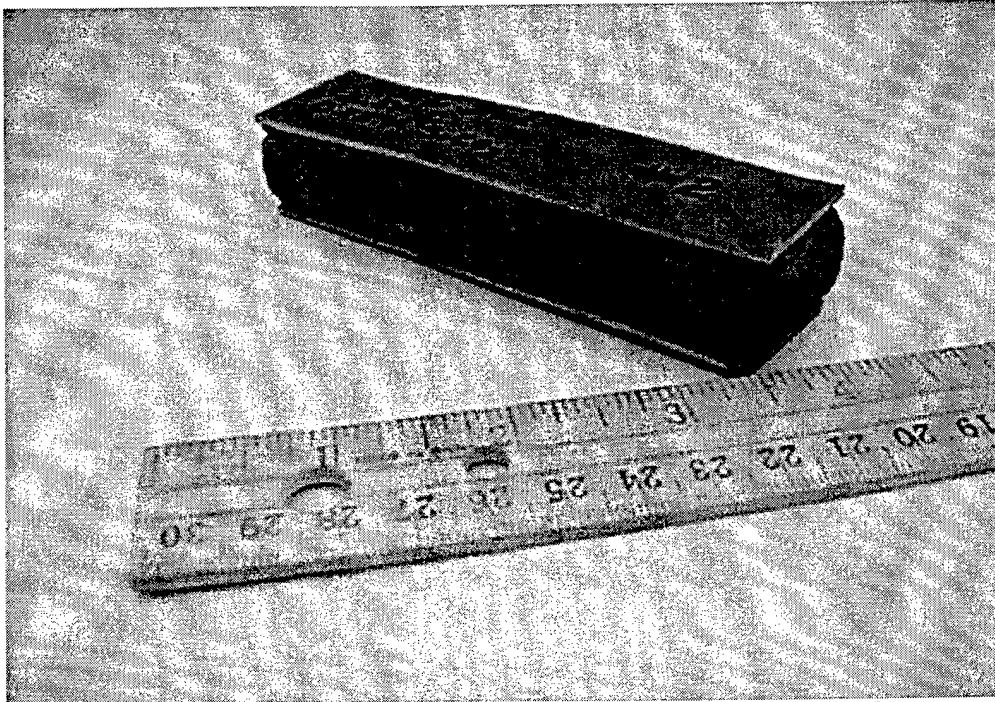


Figure 18 . Section from a carbon steel foam panel in the as-foam condition.

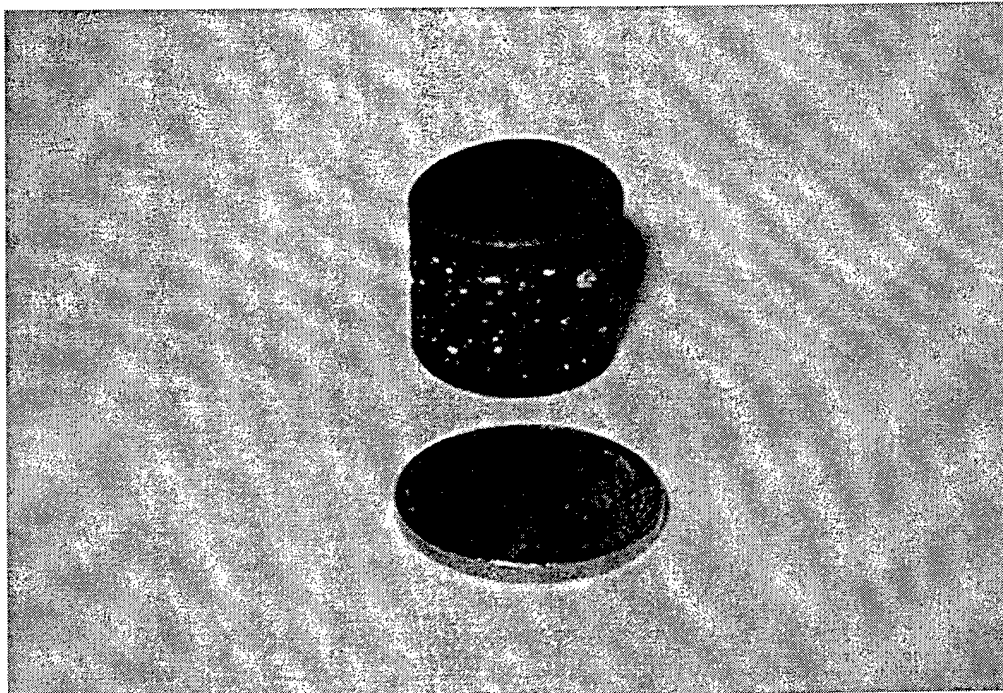


Figure 19 . 3/4 inch EDM sample cut from a 0.5% SrCO₃ HIP foam panel.

Carbon Steel Foam Panels Made by HIP plus Forging

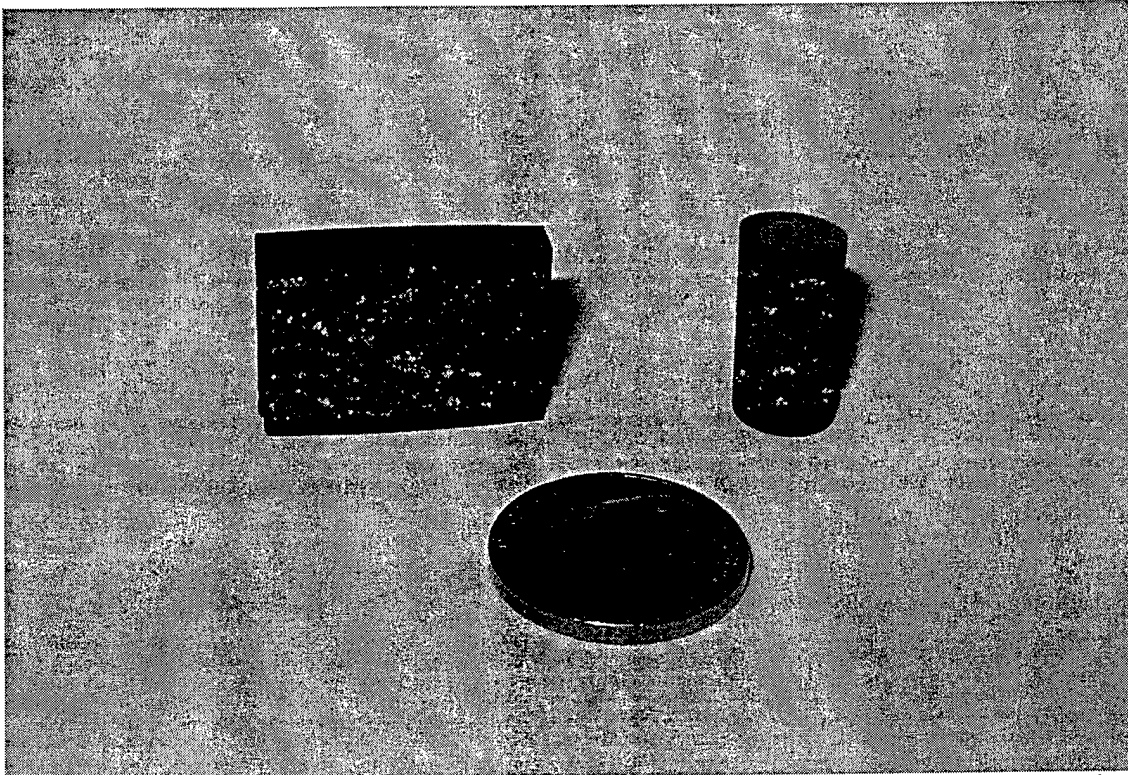


Figure 19 : Samples Wire EDM form a Steel Foam Panel Made From HIP and Forged Precursor.

In order to economically manufacturing steel foam core panels via an expected route of HIP plus rolling an experiment was conducted. A sample from HIP condition 1, that is, a 2.5% carbon plus 0.25% SrCO_3 HIP at 704°C was forged cold to 0.375 inches thick, a 60% reduction in height with the 18 gauge low carbon steel face sheets in place. Next a 1.0 inch by 2.0 inch section from the forged piece was foamed in the standard manner at about 1300°C for 4 minutes.

Small section wire EDM from this foamed panel are shown in Figure 19. The results are the best steel foam results to date. The overall density is 50% of theoretical, the low carbon steel face sheets are very well bonded, and the pore size and distribution is improved relative to the HIP and foamed route. These results are very encouraging and show that the HIP PLUS ROLLING processing route that will be proposed for phase II is viable.

Hot Isostatic Pressing of Stainless Steel Powders with Foaming Agents.

Six plate cans of stainless steel powders with chromium and molybdenum nitride foaming agents, with the schedule below, were prepared and filled in the same manner as the low carbon steel powder cans. However, these cans were HIP consolidated at higher temperature, 950°C, and were shipped to Fraunhofer for foaming.

	316L SS	2205 Dup. SS	420SS
0.5 w.% CrN	950°C/2000B/1HR	950°C/2000B/1HR	950°C/2000B/1HR
1.0 w.% Mo ₂ N	950°C/2000B/1HR	950°C/2000B/1HR	950°C/2000B/1HR

Thus far foaming results on all of the stainless steel samples have been negative. Little or no foaming has been observed thus far. An investigation is ongoing to determine the reasons for the lack of foaming in stainless to date. One possibility is that the gas escapes during foaming from the edge of the compacts. This may be due to low surface tension at the surface, perhaps because chromium oxide is volatile at the foaming temperature.

Task 2:

NDE Concepts

A meeting was held at Johns Hopkins University between UltraClad Corp. and Prof.'s Robert Green and John M. Winter to discuss NDE approaches for steel foam panels and especially with regard to future collaboration on potential Phase II SBIR as well as other programs. Results of several examples of porous samples examined by Johns Hopkins' NDE Laboratory CT Scanner were presented. While it was decided that the CT method would not be applicable as an in line manufacturing method for foam panel production, it would clearly be a valuable tool in the foam process development. The ability to 3 dimensionally map pore size distribution and morphology was judged to be outstanding. Prof. Green agreed to CT map a steel foam sample, and the results are included below.

Acquisition parameters: 160 kV @ 0.055mA
3000 views per slice
50 slices, contiguous, each 0.25mm thick

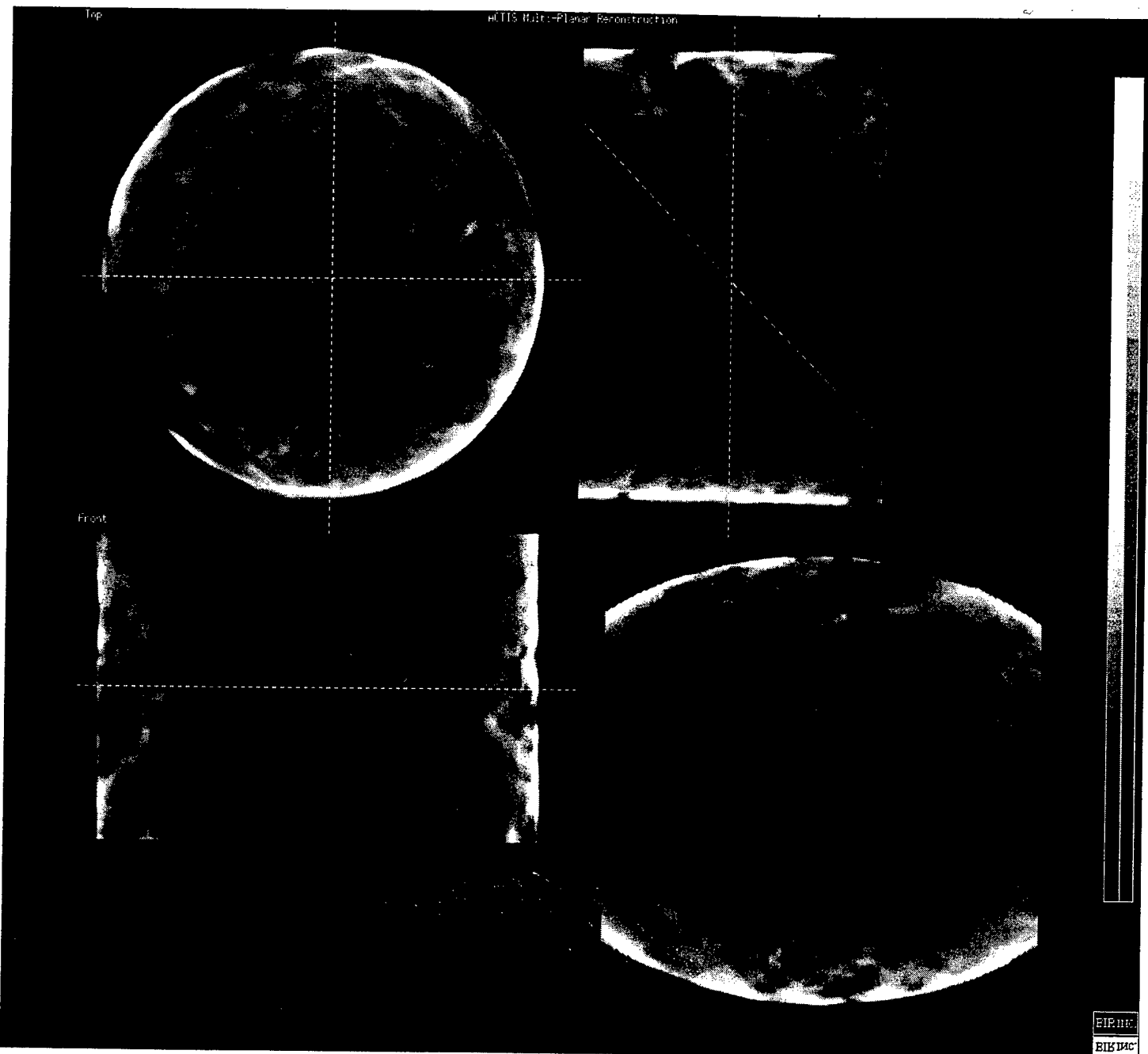


Figure 21a: CT scan of 0.75 in diameter by 0.625 inch cylinder cut from HIP carbon steel foam panel.

Multiplane Reconstruction #1

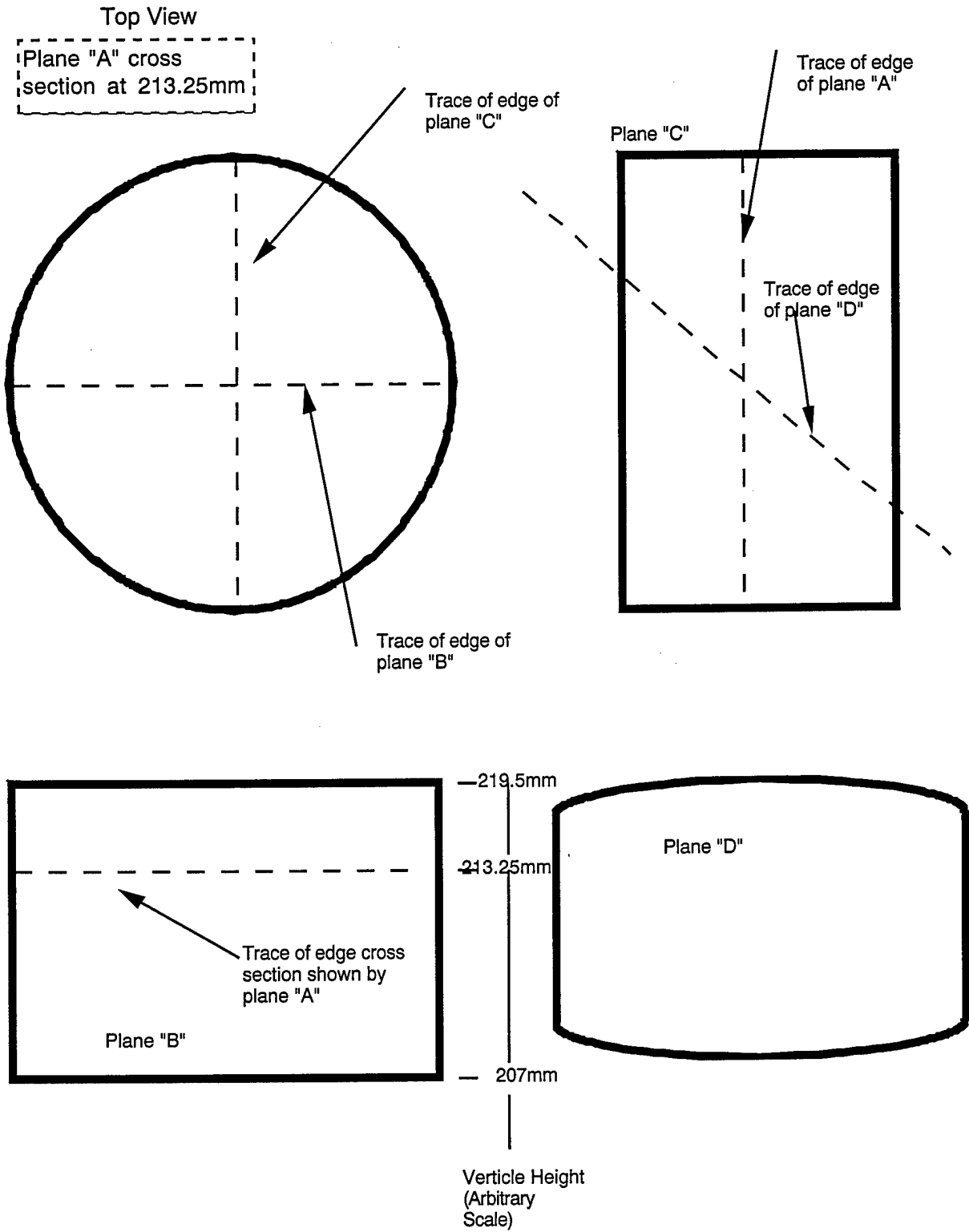


Figure 21b: CT scan of 0.75 in diameter by 0.625 inch cylinder cut from HIP carbon steel foam panel - Diagram for interpretation of 18a.

Task 3. Fabrication Methods.

No experiments were conducted on fabrication methods, however, analogs were reviewed in aluminum foam systems. Aluminum foams have been successfully joined by riveting and by laser welding.

Riveting is believed to offer the best potential for joining steel foam panels. For example, the face sheets can be riveted individually and the foam cores may be dovetailed together analogous to wood cabinet making methods.

All welding methods were judged to have the potential to destroy the foam core. However, if heat can be carefully managed the face sheets can be welded individually and foam core mechanically interlocked as above.

Task 4. Manufacturing Cost Model.

Cost data for the hot isostatic pressing production from R&D phase to production is presented below in a set of figures. Based on similar experience with other product lines cost will drop from \$150/lb to \$3.75 per lb in 100,000 production lots with yearly production in the range of 5000 tons.

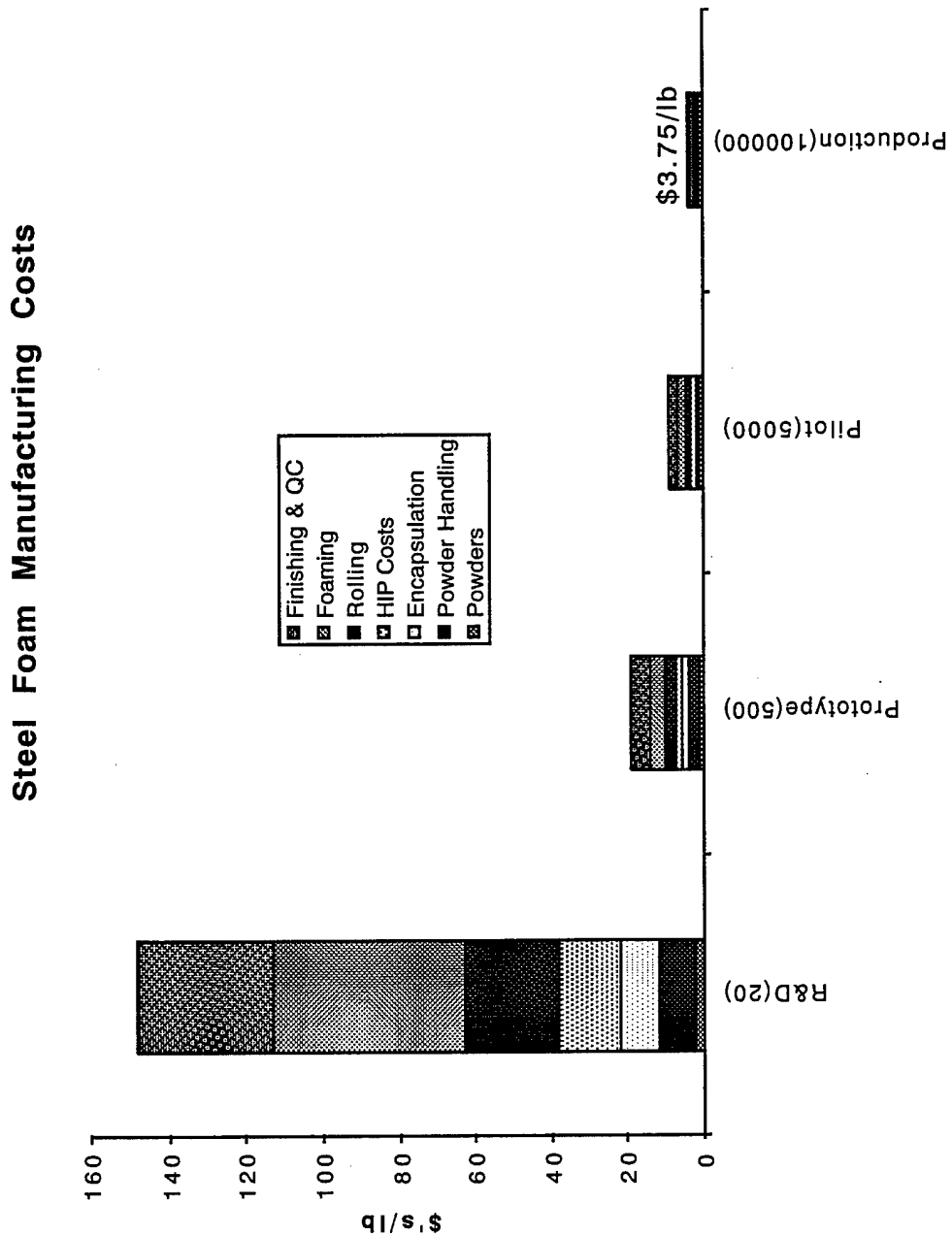
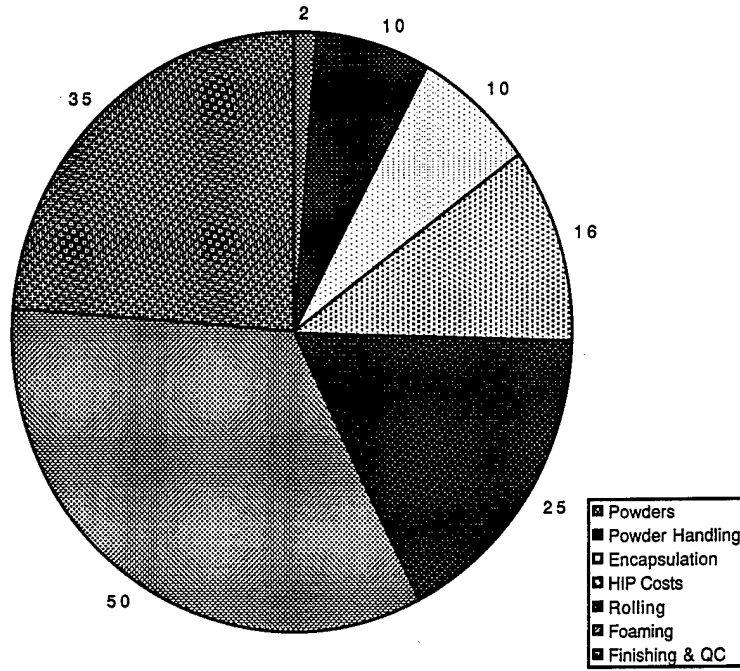


Figure 22: Product Costs for Steel Foam at Various Rates.

Steel Foam R&D Cost Breakdown



Prototype(500)

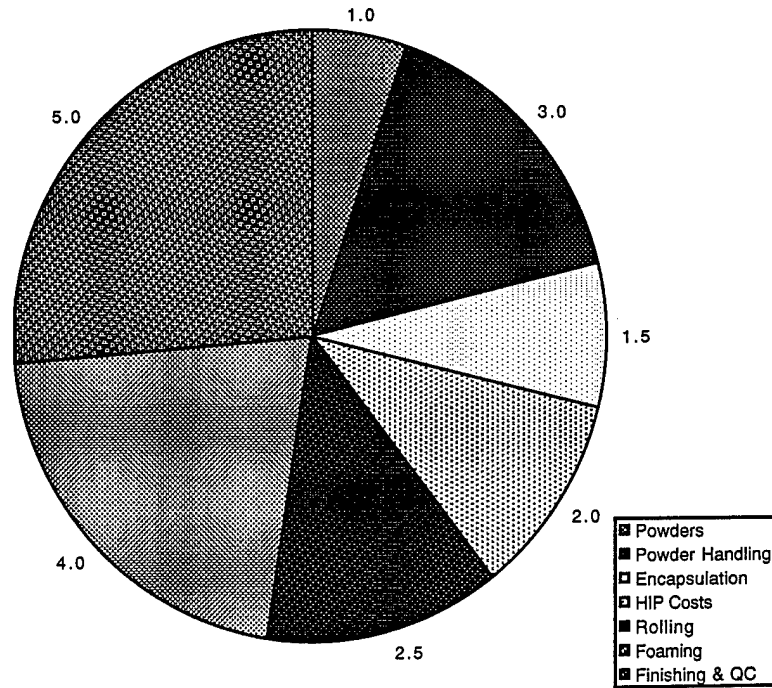


Figure 23: a) R&D Cost Breakdown-\$'s/lb, above and b) prototype cost breakdown, below. Legend in order, clockwise from 12:00.

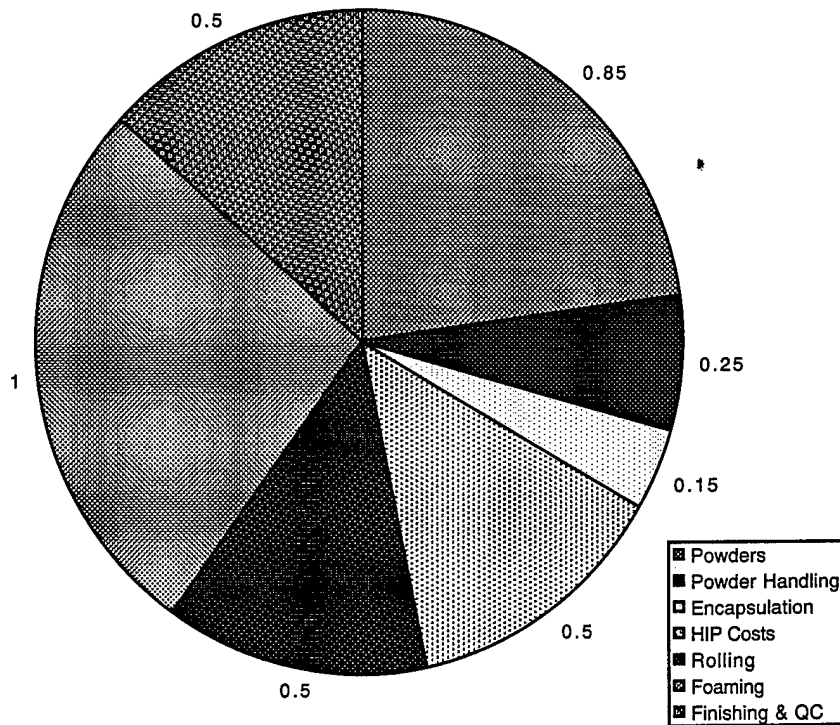
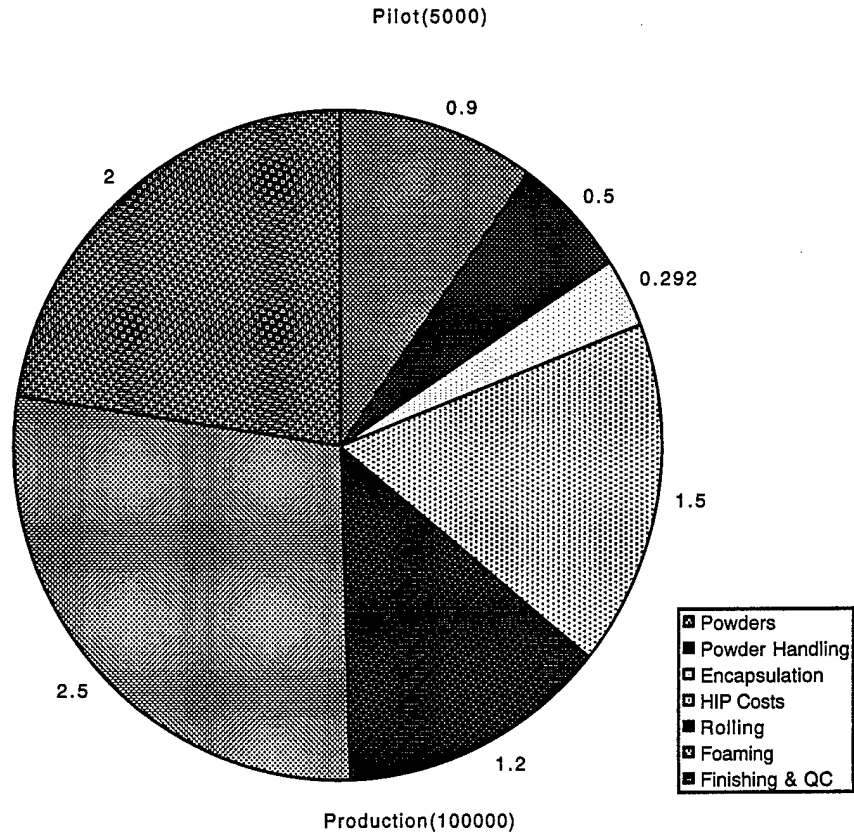


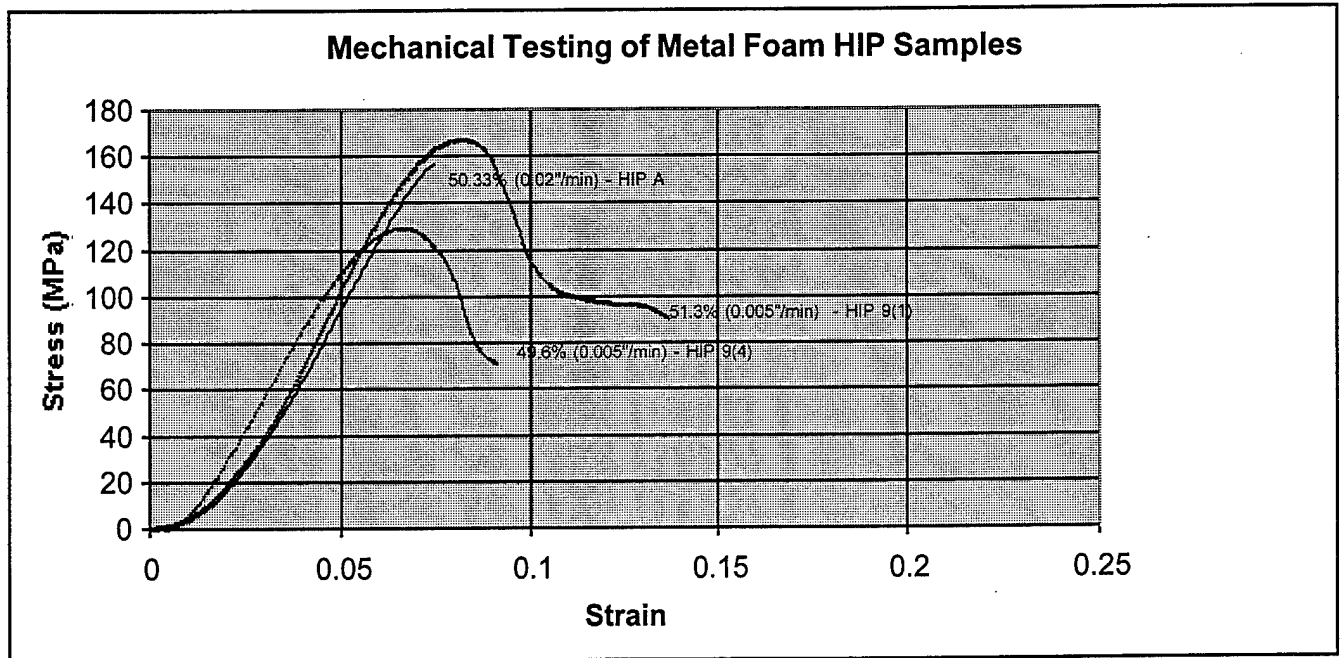
Figure 24: a) Pilot Cost Breakdown-\$'s/lb , above and b) production cost breakdown, below. Legend in order, clockwise from 12:00.

Untasked Supplemental Work

Mechanical Testing of steel Foams

Approximately 1.9 cm cubes of steel foam from sample 1-9, HIP 2.5% carbon 0.25% SrCO₃, were tested in compression, Engineering Stress versus Strain curves are shown below. The test of HIP A sample was interrupted because the load reached maximum range, a low range was used because a weaker result was expected. Two tests run at higher load ranges reached peak values of 175 MPa and 120 Mpa, results about twice as the best achieved to date with hot pressed foams even when corrected for the higher density of the HIP foam sample, 50% versus 40% of theoretical. Both tests were interrupted the curves would have continued at some time at roughly constant load. The drop in load are the result of shear band fracture across the face of the foam cubes.

Figure 25. Compressive Stress versus Strain for HIP Foam Cubes



Summary

Outstanding results have been obtained in Phase I due to the excellent teamwork between UltraClad Corporation and Fraunhofer USA, Center for Manufacturing and Advanced Materials Delaware.

Steel foams have also been produced on a laboratory scale; methods for scale-up to low cost manufacturing/fabrication via hot isostatic pressing have been investigated and demonstrated in this Phase I effort. UltraClad has made use of the Fraunhofer

USA (Delaware) foam technology via subcontract coupled with its own expertise in hot isostatic pressing, metal matrix composite technology(4,5), and scale-up of laboratory concepts to further develop steel cored foam panels. An application for a Phase II has been identified, a foam core steel door for a naval vessel constructed by Bath Iron Works.

Highlights of Phase I results include: The best steel foam density and mechanical property results to date in comparison with other steel foam processing routes. Identified problems to solve include: Understanding and control of less than ideal pore size distribution.

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