**A GENERIC APPROACH TO
IMPROVED SEMI-SOLID FORMING
OF METALS**

Final Report

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Abstract

Lack of technology for the production of large inexpensive feedstock, with uniform spherical primary phase throughout as required for semi-solid forming, has restricted realization of the full potential for the semi-solid forming process. Furthermore, narrow process windows and alloy chemistry restrictions increase process costs and limit performance attributes possible with existing semi-solid metal systems.

Successful semi-solid forming trials utilizing Chesapeake Composites Corporation's DSC™ Metals for feedstock indicate that this represents a generic approach to providing a permanent highly uniform, spherical solid phase, without electromagnetic or mechanical shearing. This approach also provides for further growth of semi-solid forming by providing for: low cost large diameter billet stock, reduced semi-solid forming costs, extension of semi-solid forming to new alloy systems, and semi-solid formed components with substantially enhanced physical and mechanical properties.

In Phase I, Chesapeake utilized DSC™ Aluminum to successfully demonstrate: the low cost production of large diameter feedstock with uniform permanent spherical solid phase, an expansion of the semi-solid forming process window, and semi-solid formed material with substantially enhanced physical and mechanical properties versus existing semi-solid forming materials.

In Phase II, the production of 6" diameter billets suitable for semi-solid forming (SSF) and the design, fabrication, and testing of SSF DSC™ Aluminum engine components will be demonstrated. Primary billet processing costs, SSF costs, heat and mass transport during SSF, and material behavior will be modeled. Basic and application specific microstructural - mechanical - thermal property databases will be developed. Finally, 4" billet SSF capability will be established and the formability map for DSC™ Aluminum above the melting point will be established.

Chesapeake Composites has met all Phase I objectives. Furthermore, Chesapeake has secured a Phase II commitment with a leading automotive OEM manufacturer, and will complete a substantial equity financing, including funds for capital equipment to be utilized in Phase II and Phase III.

In Phase I, the first objective was to demonstrate a low cost technique for the production of large diameter billet stock with the required semi-solid microstructure. Using the existing operating procedure for 3" diameter product, Chesapeake produced an aqueous ceramic slurry consisting of the dispersion phase and appropriate carrier, dispersants, binders, and fugitive filler phases to achieve the desired volume fraction of dispersion phase in the final body. The ceramic slurry was spray dried and the spray dried powder shipped to Flow Autoclave Systems Inc. for cold isostatic pressing, as in-house capability is limited to 3" diameter billets. Five green preforms were pressed with a size of about 4.5" dia. x 22" long. The preforms were returned to Chesapeake where they were sectioned into 4" long pieces and successfully burned out and fired in Chesapeake's furnaces. 4" diameter preforms were infiltrated with 99.9% aluminum using Chesapeake's inert gas pressure infiltration unit. The resultant material contained 30% sub-micron aluminum oxide with the balance 99.9% aluminum. Subsequent metallographic examination revealed a highly uniform microstructure of 30% by volume sub-micron spherical solid ceramic phase with the balance aluminum. No porosity or second phases were observed. Thus, the required microstructure for semi-solid forming of DSC™ Aluminum has been demonstrated at the 4" diameter billet size.

Chesapeake has developed a manufacturing approach for scale-up to 6" dia. billets. This approach was utilized to attract equity funding for a 5 million pound per year pilot plant for the production of 4" dia. and 6" dia. billets. The manufacturing approach is summarized as follows:

- aqueous ball milling for the production of a ceramic slurry
- spray drying for the formation of a free flowing pressable powder
- dry bag isostatic pressing for the formation of large cylindrical preforms
- burnout and firing in air kilns
- liquid metal infiltration using vertical squeeze infiltration press

All process steps utilize commercially available equipment that operates continuously or in large batches. Furthermore, the process steps are highly automated and coordinated for reduced labor costs, inventory costs and to minimize cycle time.

Finally, the results of the 4" dia. billet work and the manufacturing approach were incorporated into Chesapeake's existing cost model to evaluate the primary processing costs at the 4" dia. billet level. Results of the cost model indicate that the cost/lb. to manufacture DSC™ Aluminum billet is consistent with the market interest (what customers are willing to pay) for the product.

The second objective was to expand the process window for semi-solid forming by elimination of the need for precise temperature control and alloy compositional restrictions in the semi-solid forming process.

For the second objective Chesapeake produced several laboratory size billets, 1.5" dia. x 4" long, of two DSC™ Aluminum compositions:

- 30% Al₂O₃/99.9% Aluminum
- 40% Al₂O₃/99.9% Aluminum

These billets were machined into cylinders, 0.625" dia. x 0.790" long. An instrumented 20-ton hydraulic press with heated H-13 tool steel rams was utilized to upset the cylinders above the melting point of the metallic matrix. Using this approach Chesapeake was able to define the basic semi-solid forming process parameters including:

- material composition
- billet temperature
- die temperature
- ram speed
- ram force
- lubricant
- indirect vs. direct semi-solid forming

Finally, the microstructure of the semi-solid formed material was evaluated.

The key results of this work showed that:

- DSC™ Aluminum systems are semi-solid formable above the melting point of the matrix thereby providing for semi-solid forming of any aluminum matrix alloy system and eliminating the need for precise temperature control during semi-solid forming
- semi-solid forming was successfully demonstrated at strain rates covering 2 orders of magnitude, 10^0 s^{-1} to 10^2 s^{-1}
- semi-solid forming forces are very low, with material flow stress on the order of 10-100 p.s.i.

The third objective was to demonstrate semi-solid formed material with substantially enhanced physical and mechanical properties.

For this objective, laboratory size billets were produced using seven different DSC™ Aluminum material systems, the compositions were as follows:

- 30% Al₂O₃/1090 Aluminum
- 40% Al₂O₃/1090 Aluminum
- 50% Al₂O₃/1090 Aluminum
- 30% Al₂O₃/6061 Aluminum
- 40% Al₂O₃/6061 Aluminum
- 30% Al₂O₃/2024 Aluminum
- 40% Al₂O₃/2024 Aluminum

The nomenclature used for aluminum alloys in this proposal has greater latitude than is normally used for alloy description. To make alloy comparisons easier, the wrought designation is used for semi-solid formed samples. This nomenclature has been adopted because the matrix alloy was obtained from wrought feed-stock.

These billets were machined into cylinders, 0.625" dia. x 0.790" long. A tool steel die for the production of a 0.25" dia. x 2.25" long rod was fabricated and utilized in conjunction with the 20 ton press to semi-solid form each of the above compositions utilizing the process parameters developed previously. The semi-solid formed material was tensile tested at Westmoreland Mechanical Testing & Research and the elevated temperature strength evaluated using hot hardness testing at Chesapeake. The hot hardness test used was Rockwell 'B', 1/16" diameter tungsten carbide ball, which is the standard for room temperature testing. The hardness indents for a given material were all made on the same test sample. To reach the elevated temperatures, the sample was heated to increase its temperature 100°C in 15 minutes, after which the temperature was held for 30 minutes before indents were made. At elevated temperature, the applied load was reduced to maintain the indent diameter within the accepted bounds for Rockwell hardness testing. The hardness measured at room temperature is consistent with the tensile strength measured on similar samples. Metallographic examination of the semi-solid formed materials was performed. Finally, the performance and microstructure of the semi-solid formed material was compared to their cast or wrought counterparts.

The key results of this work showed:

- SSF DSC™ Aluminum exhibited good die fill and surface finish, as shown in Figure 1.
- further verification of the elimination of precise temperature control and freedom from matrix alloy chemistry
- very good mechanical properties (yield strength, UTS and ductility) were observed in the semi-solid formed DSC™ Aluminum systems versus wrought monolithic and SSF 356 systems, as shown in Figures 2 through 4.
- DSC™ Aluminum systems offer substantially improved elevated temperature performance as shown in Figures 7 through 11, substantially improved elastic modulus Figure 5, and substantially reduced CTE as compared to the monolithic wrought counterparts as shown in Figure 6 for the matrix alloys
- the microstructure of the semi-solid formed DSC™ Aluminum systems exhibited a highly uniform alumina phase, no porosity or reaction phases in any of the systems
- the microstructure of the semi-solid formed material exhibited homogenization and grain refinement versus the billet stock

Figure 1. Photograph of DSC™ Aluminum (2024-T4 / 30% Al₂O₃) semi-solid formed bar (~2.5" long x ~ 0.25" dia. with 0.625" dia. head to feed shrinkage) used for testing. Note good die fill and surface finish with fine flashing at parting line.

Figure 2. Bar chart showing room temperature yield strength for three DSC™ Aluminum semi-solid formed materials (this work) compared with their monolithic wrought matrix alloys (handbook data) and recently published data for SSF A356. Note that the SSF DSC™ Aluminum systems offer substantially improved yield strength versus their monolithic wrought matrix alloys and all are greater than SSF A356-T4.

Figure 3. Bar chart showing room temperature ultimate tensile strength for three DSC™ Aluminum semi-solid formed materials (this work) compared with their monolithic wrought matrix alloys (handbook data) and recently published data for SSF A356. Note that the SSF DSC™ Aluminum systems offer substantially improved ultimate tensile strength versus their monolithic wrought matrix alloys and all are greater than SSF A356-T4.

Figure 4. Bar chart showing room temperature tensile elongation for three DSC™ Aluminum semi-solid formed materials (this work) compared with their monolithic wrought matrix alloys (handbook data) and recently published data for SSF A356. Note that the SSF DSC™ Aluminum systems offer ductility up to 5% in the SSF (cast), T4 condition with 30 volume percent alumina particles.

Figure 5. Bar chart showing room temperature Young's modulus for three DSC™ Aluminum semi-solid formed materials (this work) compared with their monolithic wrought matrix alloys (handbook data) and recently published data for SSF A356. Note the substantial increase in modulus offered by SSF DSC™ Aluminum compared to wrought matrix alloys. The modulus of 1090-30%-SSF is low due to rounding of the tensile curve.

Figure 6. Bar chart showing thermal expansion for three DSC™ Aluminum semi-solid formed materials (this work) compared with their monolithic wrought matrix alloys (handbook data) and recently published data for SSF A356. Note that SSF DSC™ Aluminum offers reduced thermal expansion compared to the other materials shown.

Figure 7. Elevated temperature hot hardness for two DSC™ Aluminum semi-solid formed materials (30% and 40% alumina) based on a pure aluminum (99.9%) matrix compared to the monolithic aluminum matrix alone. Note that the wrought matrix alloy designations are maintained for the SSF systems to facilitate easy recognition of the alloys.

Figure 8. Elevated temperature hot hardness for two DSC™ Aluminum semi-solid formed materials (30% and 40% alumina) based on a 2024-T4 aluminum matrix compared to the monolithic aluminum matrix alone. Note that the wrought matrix alloy designations are maintained for the SSF systems to facilitate easy recognition of the alloys.

Figure 9. Elevated temperature hot hardness for two DSC™ Aluminum semi-solid formed materials (30% and 40% alumina) based on a 6061-T4 aluminum matrix compared to the monolithic aluminum matrix alone. Note that the wrought matrix alloy designations are maintained for the SSF systems to facilitate easy recognition of the alloys.

Figure 10. Elevated temperature hot hardness comparison of DSC™ Aluminum semi-solid formed materials with three different matrix alloys and 30% sub-micron alumina particle volume fraction. Note that the wrought matrix alloy designations are maintained for the SSF systems to facilitate easy recognition of the alloys.

Figure 11. Elevated temperature hot hardness comparison of DSC™ Aluminum semi-solid formed materials with three different matrix alloys and 40% sub-micron alumina particle volume fraction. Note that the wrought matrix alloy designations are maintained for the SSF systems to facilitate easy recognition of the alloys.