FY 2018, Phase II, Release 2, SBIR topic 13a SOLAR – Next Generation CSP Components for High Temperature Molten Chloride Salt Development

High Toughness Cermets for Molten Salt Pumps

Program Summary

Research Objective

The DOE seeks major reductions in the cost of concentrated solar power (CSP) to achieve subsidy-free, cost-competitive solar power by 2020. A significant step in achieving lower cost is the evolution to higher temperature operation. Molten chloride salts, and in particular KCl-MgCl2 currently appear to be to be the materials of choice to operate at these temperatures. The high working temperature and corrosive properties of these salts present an aggressive environment for CSP components. Valves seat and seals, bearings and impellers for pumps have additional requirements because of the dynamic mechanical loads that these components must survive. Alloys do not have good compatibility with salt and are expensive. Ceramic composites are difficult to form. This project offers a proof of concept for the use of ceramic, metal matrix nanocomposite materials to enable the economical construction and operation of molten salt pumps.

Research Completed

1. Several grades of hierarchically structured ceramic-metal nanocomposites (cermets) were compounded, molded and evaluated.
2. Measured corrosion resistance of cermets in KCl–$MgCl_2$ salt at 750ºC.
3. Measured the sensitivity of the metal matrix ceramic nanocomposite sintering temperature to corrosion resistance, hardness and density.
4. Implemented a bench test methodology for wear properties of materials in molten salt.
5. Estimated the volume production costs of typical bearings.
6. Built working relationships with molten salt pump suppliers and the system supply chain.

Research Findings

All of the objectives of the program were met.
Initial corrosion tests confirm acceptable corrosion resistance for the CSP Gen 3 application. Mechanical tests confirm the predicted high hardness and toughness at room temperature. The results of this project confirm the potential to use HybriMet cermet as the material of construction for bearings, seals and impellers for CSP molten 750ºC KCl-MgCl2 or MgCl2:KCL:NaCL chloride salt pumps. The first pin-on-disk tribometer capable of running in a high temperature molten salt environment was commissioned. This capability will accelerate the selection of bearing materials for high temperature salt applications and advance the understanding of wear mechanisms in molten salt. Calculations confirm that cermet is more economical than many super alloys. A major pump manufacturer has agreed to collaborate to design and build a laboratory salt loop pump using Powdermet's HybriMet materials and to provide a quotation for a pump for the CSP Gen 3 demonstration.

Applications

- Bearings, seals, impellers, coatings for CSP molten 750ºC chloride salt pumps.
- Commercial and industrial pumps in extreme environments like subsea oil field pumps.
- Wear resistant bearings in lightly lubricated and product lubricated pumps and engines.

Phase I Final Report

Contract Number: DE-SC0017677
FY 2018, Phase I, Release 2, SBIR topic 13a SOLAR – Next Generation CSP Components for High Temperature Molten Chloride Salt Development

High Toughness Cermets for Molten Salt Pumps

Contract Period: September 9, 2017 to June 11, 2018

Contractor
Powdermet Inc.
24112 Rockwell Dr.
Euclid, OH 44117

Program Manager [redacted] [redacted]	Technical Monitor [redacted]
Principal Investigator [redacted]	Alternate Contact [redacted]

Identification and Significance of the Problem and Technical Approach

The DOE seeks major reductions in the cost of concentrated solar power (CSP) to achieve subsidy-free, cost-competitive solar power by 2020. A significant step in achieving lower cost is the evolution to higher temperature operation. Higher temperature energy conversion allows for much better thermodynamic efficiency. The Solar Energy Technologies Office (SETO) has targeted 700ºC and higher operating temperatures for the next generation of CSP. Molten chloride salts, and in particular $KCl\text{-}MgCl_2$ currently appear to be to be the materials of choice to operate at these temperatures. The high working temperature and corrosive properties of these salts present an aggressive environment for CSP components. Valves seat and seals, bearings and impellers for pumps have additional requirements because of the dynamic mechanical loads that these components must survive. This proposal address this need for improved materials for active mechanical components in molten salt. Over the last few years, some data on the corrosion of materials in chloride salts, $KCl\text{-}MgCl_2$, has been generated by the University of Wisconsin, Savannah River National Laboratory and some others. The recent materials of interest are metal alloys and ceramic composites.[1] Alloys are capable of being formed into piping and other components, have reasonable strength up to 1000ºC, have reasonable compatibility with salt, but are expensive. Ceramic composites have good high temperature properties and salt resistivity but are difficult to form.

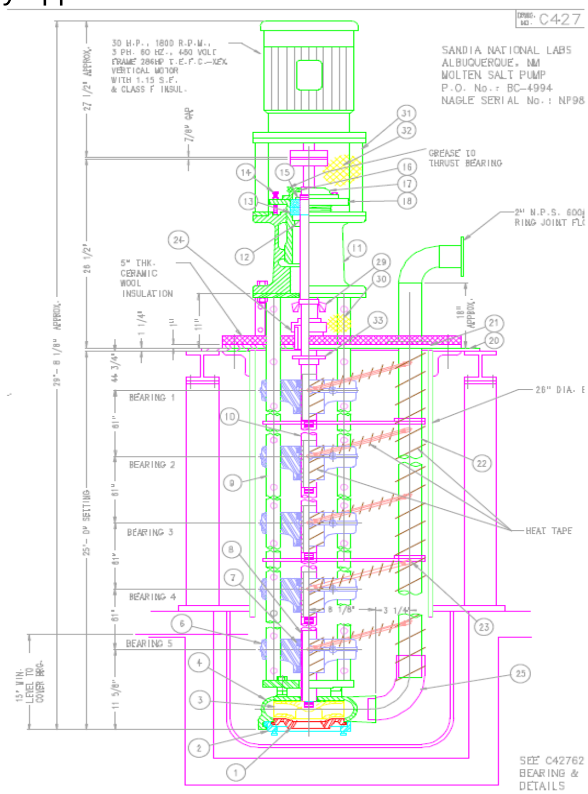


Fig. 1 Salt Lubricated Pump[3]

Corrosion in molten salts is due to impurities, transport rates and activity differences. Plate out of elements from hot sections onto cold sections of flow systems is an important consideration. Corrosion is a complex electrochemical process where the molten salts attack the most active alloying additions in the metal.[2] Possible ways to mitigate the corrosion are through salt chemistry control and material modifications. In the salt, this entails cleaning the salt of water, oxygen and contaminants and elemental additions for redox control. In the materials we can consider improved alloy chemistry, surface treatments and grain boundary engineering.[2]

The rate of corrosion is proportional to the electrochemical activity of the elements and the alloy composition. Alloys containing Cr exhibit Cr dissolution from the alloy that is higher in the alloys containing more Cr. The dissolution of Cr is preferential at the grain boundries.[7] Pure nickel, Ni-201, consistently exhibits lower weight loss.[2]

Most of the specialty alloys under consideration have sufficient high temperature strength to operate at 800ºC, but drop off substantially at 1000ºC.[1] Haynes 230 is best, having an Ultimate tensile strength of about 700 MPa at 1000ºC but it has a high chrome content, 22% and therefore the higher corrosion rates.

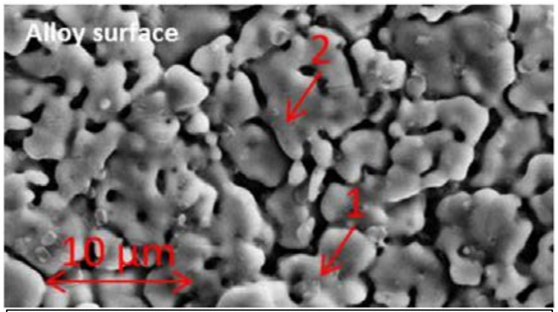


Fig 2. Haynes 230 surface after exposure to KCl-$MgCl_2$ at 550ºC for

Composite technology offers a way to engineer tensile strength and hardness in a metal, either pure or an alloy, independent of the metal composition. The metal or alloy can be compounded for corrosion resistance, processability, moldability and then reinforced by the composite fillers. Pure nickel, which has a tensile strength of about 180 MPa can be reinforced by ceramic nanoparticles to achieve a tensile strength as high as 1032 MPa.[4, 5] Separating the strength from the metal properties, and the choice of ceramic type and concentration in the composite enables the material to be tailored for thermal expansion, corrosion resistance, etc. This provides a platform technology that can be manipulated by materials engineers to solve problems that are very difficult today. Zero contiguity in the metal matrix ceramic nanocomposite is key to achieving high strength and toughness. Zero-contiguity refers to the property of a two phase composite material where no two particles of the discrete constituent are in contact with one another. Powdermet's unique process achieves the near zero contiguity goal.[4, 5] Powdermet has trade named this material HybriMet™.

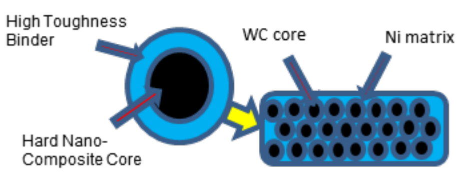


Fig 3. Metal matrix ceramic nanocomposite concept

HybriMet™ is a family of hierarchically structured ceramic-metal (cermet) nanocomposites with superior wear resistance, lower friction, higher fracture toughness, and improved reliability and durability compared to conventional bearing materials including tool steels and ceramics. HybriMet offers the frictional and life benefits of ceramics such as Si_3N_4, but with the toughness and ease of manufacture and reliability of steel.

The structure combines high hardness nanocomposite zones with tough ductile zones in an engineered manner. The hard ceramic core can be virtually any nitride or carbide (such as Titanium Nitride or Tungsten Carbide).
In prior research, to address bearing life in severe environment, highly loaded, lubrication starved and contaminated applications, Powdermet demonstrated a very wear resistant, near zero-contiguity nanocomposite having hardness near silicon nitride (13.7 GPa for NiTiN and 14.7 GPa for NiWC) and toughness like 52100 chrome bearing steel (29.5 $MPa/m^{1/2}$ for NiWC)[4, 5]. Powdermet's nanocomposite consists of metal and ceramic where the metal matrix used has been either cobalt (Co) or nickel (Ni) and the ceramic is titanium nitride (TiN) or tungsten carbide (WC). Powdermet hierarchically engineers the nanocomposite structure by coating nano-particles of ceramic with an appropriate

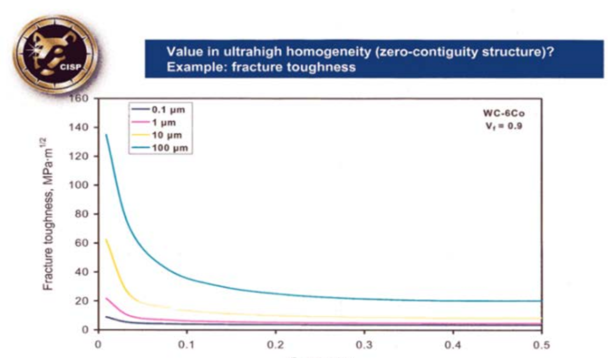


Fig 4. Zero contiguity structures significantly increase properties of metal ceramic

metal alloy, coating this particle with the metal matrix and then compacting and sintering the material in a controlled process to carefully control grain growth.

The composite material has mechanical properties higher than tool steel (29.5 $MPa/m^{1/2}$ toughness). The nickel matrix reinforced with WC composite ([redacted] has a room temperature tensile strength of 944 MPa, higher than either Cartech L-605, Haynes 230, Hastelloy C276.

HybriMet NiWc Typical Properties

Property	Method	Conditions	Units	Typical Value[1]
Density	ASTM C134	RT	g/cc	11.75
Hardness	ASTM E18	RT	GPa	14.93
Vickers Hardness			HV (300)	1521
Rockwell Hardness	ASTM E18	RT	HRC	61
Tensile Strength	ASTM E8	RT	Mpa	944
			Ksi	137
Tensile Elongation	ASTM E8	RT	%	0.25
Elastic Modulus	ASTM E8	RT	GPa	352
			$x10^6$ psi	51
Flex Strength	ASTM C1161	RT	Mpa	761
			Ksi	110
Compressive Strength	ASTM E9	RT	Mpa	1467
			Ksi	213
Compressive Modulus	ASTM E9	RT	Gpa	352
			$x10^6$ psi	51
Bulk Fracture Toughness	ASTM C1421	RT	$Mpa*m^{\wedge 1/2}$	29.5
Coefficient of Friction	pin-on-disk	RT, dry		0.44
Coefficient of Friction	pin-on-disk	RT, lubricated		0.14
Wear Rate	pin-on-disk	RT, lubricated	$mm^3/N\text{-}m$	$<< 10^{-12}$
Rolling Contact Fatigue		RT, 2.5 Gpa	cycles	$> 60x10^6$
Thermal Conductivity	ASTM E1461	0 to 500ºC	W/m-ºC	30
			$BTU\text{-}in/hr\text{-}ft^2\text{-}ºF$	208
Specific Heat Capacity	ASTM 1461	0 to 500ºC	J/g-ºC	0.23
Thermal Diffusivity	ASTM 1461	0 to 500ºC	cm^2/sec	0.11
Electrical Conductivity				high
Thermal Expansion	ASTM E228	0 to 750ºC	ppm/ºC	7.97

The demonstrated nanocomposite materials offer very low friction when tested with a minimal amount of poorly lubricating fluids, and are extremely erosion and wear resistant, offering erosion and wear resistance similar to diamond like carbon and CrN coatings (in air) Tribological tests conducted during this earlier research, showed extremely low wear in both dry and in lubricated environments[4, 5], encouraging continuing development as rolling elements in ball and roller bearings and as plain bearings. When tested dry, without any lubricant, the coefficient of friction is 0.5.

The material is consolidated using spark plasma sintering (SPS). This process allows molding the material with very controlled grain growth. Because the cycle can be run without melting the metal matrix, the dispersion of the ceramic phase can be maintained and the ceramic elements do not alloy with the metal matrix. This preserves the engineered hierarchical structure of the composite.
The cermet is expected to be economical and lower cost than specialty metal alloys for molten salt applications. First, because nickel performs best in corrosion tests to date, we expect to validate in this project substantially longer life in $KCl\text{-}MgCl_2$ salts. We also expect this benefit to extend to other chloride molten salts as requirements evolve.

Specialty alloys under consideration cost $50 to $1000/kg for stock (in China) and upwards of $1000/kg for finished parts. Molded billets of NiWC composite, now in low volume development quantities cost $650/kg. Scale up and molding efficiencies will drive down this cost to about $200/kg. The CVD coating of the ceramic powder is currently run at the rate of several tons per year, so the production costs of the basic component of the raw materials, in this volume, is well understood. The composite is readily machineable using EDM and grinding techniques. **Most importantly, no complex and costly heat treating is necessary to attain the composite's properties.** Future development (beyond this Phase I project) on molding complex shapes and extrusion can further reduce this cost.

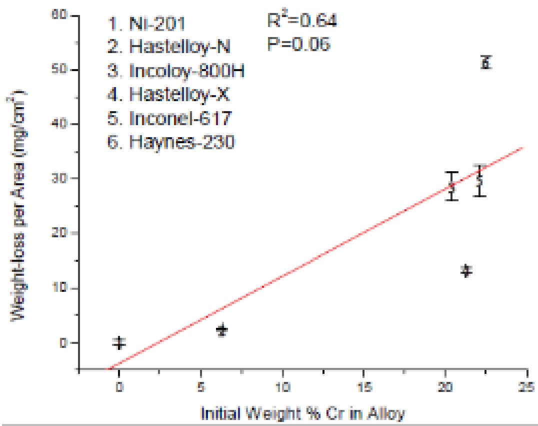


Fig 'orrelation between Weight Loss and initial Cr Content of allov[2]

Existing data on the corrosion rate as measured in weight loss per unit area of nickel is 55 times less than that of Haynes 230 and 30 times less than Incoloy 800H.[4,6] See Figure 6. Assuming the nickel composite achieves the same corrosion resistance as the nickel tested, and the material cost is about the same as the specialty alloy, then the lifetime cost of composite part, ignoring the benefits of reduced downtime and maintenance, is in the range of 2% to 4% of the specialty metal alloy.

The zero contiguity metal matrix ceramic nanocomposite is a platform technology the offers economy and flexibility to engineer the coefficient of expansion and material properties for a variety of parts, salts and temperature environments.

This project addresses another industry need. Today there is no efficient bench test for the initial evaluation and selection of materials for bearing wear in molten salt. We intend to establish a test methodology for wear in molten salt. This capability when established, will substantially accelerate the development and qualification of candidate materials. In this project, we will modify the University of Wisconsin impeller test fixture (essentially a rotating disk in a salt bath) to include a weighted pin that will ride on the disk. This first attempt at a pin on disk in molten salt test fixture will be a rudimentary example of the standard tribological pin on disk test. We will include in our Phase II proposal the further development of a fixture for journal bearing tests in molten salt.

Objectives

SETO CSP program seeks major price reductions. Higher temperature energy conversion allows for better thermodynamic efficiencies. The proposed cermets promise long bearing life in molten salt over 700ºC. Zero contiguity cermets provide the opportunity to reduce the cost of specialty metals and metal alloys by compounding them with inexpensive ceramics. The development of bench tests for molten salt bearings will reduce the cost and shorten the time for development of molten salt compatible bearings.

Powdermet expects to make molten salt pump bearings, seals and impellers, or blanks for these components and sell them to pump manufactures like Sulzer and Flowserve. Powdermet also expects to develop a salt bearing bench test methodology that will accelerate the qualification of materials for the CSP Gen 3 program. Upon success in this program, Powdermet see evolution to the application of the material technology to pipe and other molten salt components.

Success in this project will eliminate a barrier in the path to realize cost competitive solar power that can be dispatched 24/7. This product is based upon the successful anti-corrosion coatings developed by Powdermet. We believe that the product has a high probability of commercial success.

Our specific objectives for this program were to:

1) Validate the application of ultra-high toughness hierarchically designed ceramic-metallic nanocomposite materials for molten metal pumps, seals, and bearings.
2) Demonstrate high corrosion resistance of zero contiguity cermets in KCl –$MgCl_2$ salt at 750ºC.
3) Evaluate the sensitivity of the metal matrix ceramic nanocomposite sintering temperature to corrosion resistance and physical properties.
4) Develop and implement a bench test methodology for wear properties of materials in molten salt.
5) Estimate the volume production costs of typical components in zero contiguity cermet.
6) Build working relationships with molten salt pump suppliers and the system supply chain.

Summary Research Accomplishments and Findings

All of the objectives of the program were met.

Initial corrosion tests confirm acceptable corrosion resistance for the CSP Gen 3 application. The NiWC3 showed very good performance with essentially no weight change, no apparent nickel layer and no apparent attack on the surface. Mechanical tests confirm the predicted high hardness and toughness of the cermet at room temperature. The results of this project confirm the potential to use HybriMet cermet as the material of construction for bearings, seals and impellers for CSP molten 750ºC KCl-$MgCl_2$ or MgCl2:KCL:NaCL chloride salt pumps. The first pin-on-disk tribometer capable of running in a high temperature molten salt environment was successfully commissioned. This capability will accelerate the selection of bearing materials for high temperature salt applications and advance the understanding of wear mechanisms in molten salt. Calculations show that cermet is more economical than super alloys. A major pump manufacturer has agreed to collaborate to design and build a laboratory salt loop pump using Powdermet's HybriMet materials and to provide a quotation for a pump for the CSP Gen 3 demonstration.

Measurements show NiWC toughness (29.5 MPa/m1/2) is 1.5 times higher than 52100 chrome bearing steel and compressive strength (1467 MPa) is 2 times higher than Silicon Nitride. Earlier tests demonstrated these nanocomposite materials offer very low friction when tested with a minimal amount of poorly lubricating fluids, and are extremely erosion and wear resistant, similar to diamond like carbon and CrN coatings (in air). The 250 hour, 750ºC KCl-MgCl2 static immersion tests have verified corrosion resistance near that of pure Ni and better than Haynes 230.

Technical Approach and Results

Work Plan

Based on the technical objectives, a work plan was developed to produce the desired advancements in bearing and wear components in molten salt. The work plan was focused on the validation of the application of ultra-high toughness hierarchically designed ceramic-metallic nanocomposite for molten metal pumps, seals, and bearings. This program tested these materials in CSP molten salt environments to verify their performance, leading to fabrication of low maintenance, durable, and reduced energy (through reduced friction) pumps for CSP systems.

The work plan focused on NiTiN and NiWC nanocomposites, developed in previous research,[4, 5] the fabrication of test specimens, the measurement of corrosion rates and comparison to commonly considered metal alloys, the measurement and analysis of mechanical properties, and a manufacturability assessment. Working relationships were built with salt pump manufacturers. Additionally, this program established a methodology for accelerated bench testing candidate materials for tribological properties in molten salt.

Work plan and approach modifications

During the program, it has become apparent that to provide a useful pump solution, we need to expand the project beyond the molded components offered in this Phase I. For pump manufacturers to be able to quote and supply a CSP Gen 3 demonstration pump, they need a solution for all of the pump components. In Phase II and CSP Gen 3, we propose to develop a process to flame spray HybriMet cermet to coat pump shafts, housings and other components, providing a whole pump solution.

Because of the delayed award of the grant supporting this project we had to accelerate the tasks to complete the work in 9 months rather than the originally planned 12 months. To accomplish this schedule acceleration we decided to:

i) Utilize NiWC and NiTiN billets previously molded
ii) Compound and mold only the new candidate materials, NiWC2 and NiWC3 and time at temperature specimens.
iii) Use NiWC (T1) mechanical data being generated under a corporate sponsored research contract at the time of this award
iv) Select the second primary material candidate, NiTiN (T1) for mechanical data measurement immediately at the beginning of this award.
v) Reduce the time period of the salt immersion tests from 500 hours to 250 hours.
vi) Select HybriMet NiWC and Haynes 230 as the materials to test in the wear rig.

The forest fires in California last fall caused the molding of our billets to be delayed 4 weeks resulting in a 4 week delay in starting our static immersion salt corrosion tests. The main resolution was to reduce the corrosion test time from 500 hours to 250 hours. This change enabled us to obtain our preliminary results in February 2018.

Task 1. Fabricate Test Specimens

This task supports Objectives 1, 2 and 3. The powder preparation was done at Powdermet's pilot plant in Euclid Ohio (experts in CVD coating and metallurgy), the consolidation at Thermal Technologies in Santa Rosa CA (developers and suppliers of SPS equipment) and the machining at shops local to Powdermet. Powdermet personnel visited the local machine shops and conducted regular phone reviews to manage the tasks. The base raw material, the CVD coated ceramic, was prepared using the current production process. While in past research we have used both Ni and Co metal as the matrix material, here only used Ni. The electrochemical potentials and the existing data indicate that Ni is the better choice for low salt corrosion and it will reduce the potential for transport corrosion from or to other system components. Powdermet has prepared in the past several grades of the metal coated ceramic. For this project, we have chosen three grades of coated tungsten carbide (WC) and one grade of titanium nitride (TiN). These grades correspond to proven grades of Powdermet's PComP anti-corrosion coatings. Therefore although the metal matrix will, in this project, be nickel, the interface coating on the ceramic will contain other metals. The volumetric composition in each material is about 40% metal and 60% ceramic (either TiN or WC). The nickel metal matrix was dry blended with the metal coated ceramic.

The compounded powder was molded using spark plasma sintering (SPS). SPS is a process for densifying nanosize ceramics, metals, and composites. An electric power load on a powder heats it by direct joule heating along with conductive heat from a graphite mold. The current may be up to several tens of thousands of Amperes and is very effective in quickly heating samples. The whole SPS process is so fast that crystalline grains do not have time to grow, producing consistent nanosize grains and high-density product. Today the power sources have expanded to include AC, pulse DC, pulse duration DC and their combinations, allowing the electrical field to produce more reactive effects at the interface between powder particles. For example, the electrical field may cause a spark between metal particles to remove the oxide layers from surfaces or spark plasma between particles to produce a local high temperature. A key factor is the proper use of mechanical pressure to make processing fast and to prevent the building of neck formations at the initial stage of sintering. The mechanical pressure on samples closely interfaces the crystalline grains. The interface and crystal diffusions are much slower than surface diffusions so that the grains grow slowly while removing porosity during the densification stage.[8]

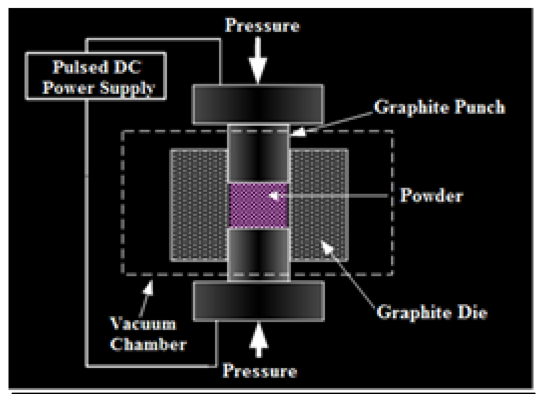


Fig 8. Schematic of SPS System

The SPS molding process for this composite has been developed and the parameters set for simple geometries; process parameters for complex geometries and larger parts still need development. The molding temperature and the time at temperature impact the material grain growth. To explore the grain size and its impact on the corrosion results, we molded the NiTiN and NiWC materials at two temperatures with a 50ºC spread at one hold time. The sensitivity of the corrosion rate to the molding temperature is expected to be useful in future material optimization.

The molded disks were wire EDM cut into 10 mm X 30mm X 0.1mm specimens for the corrosion testing and appropriate specimens for the mechanical property tests. The corrosion specimens were surface ground to bearing grade finish to facilitate corrosion measurements.

Task 2. Salt Corrosion

Demonstrate high corrosion resistance of zero contiguity cermets in KCl –$MgCl_2$ salt at 750ºC.

A series of different cermet samples (NiTiN, NiTiN/T2, NiWC, NiWC2, NiWC3 and NiWC/T2) were all tested in triplicate, exposed to purified and reduced MgCl:KCL at 750^{o}C for a period of

250 hours. A Haynes 230 sample was exposed in the same salt to serve as a standard to compare the corrosion rates between this known chemically resistant alloy and the cermets. This Haynes 230 standard was also placed in triplicate in the same pot for the same time duration as the cermets.

Initial post exposure analysis of the samples showed good corrosion resistance with just a slight change in the apparent surface roughness from the 800 grit polished unexposed sample and the exposed sample. The change in the surface of the cements was found to be similar to that of the Haynes 230 for all cases (see figure). Weight and dimensional analysis of the sample showed negligible change for most of the samples with the NiWC and NiWC3 performing very similar if not better than the Haynes 230 while the NiTiN samples realized a weight loss versus a weight gain but still within acceptable limits (see weight loss per unit surface area averages below). Cross sectional SEM and optical images were also taken which showed a typical corrosion layer on the Haynes 230 (with some chrome depletion at the surface and Cr migration to the grain boundaries) consistent with previous testing. For the cermets it appears that there is very little attack on the base metal. The NiWC sample exhibited a thin nickel layer that formed on the surface of the sample. The SEM and optically images could both clearly see the structure of the coated WC particles and there did not appear to be any significant attack or change in these WC particles (see images below). Additional line scans and EDS maps of the compositions verified the nickel surface layer on the NiWC and composition of the cermet material. There was some porosity observed in the NiTiN material.

Sample weight analysis

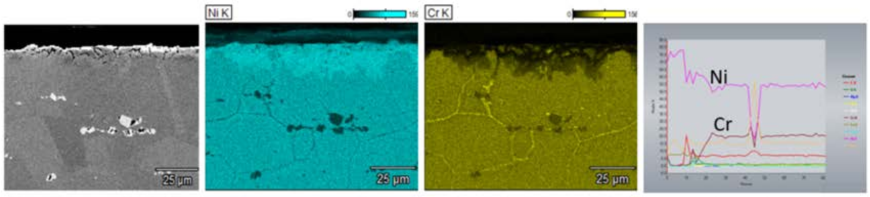
Sample Type	Sample #	Average weight change [g/mm^2]
Haynes 230	1	1.07096E-05
	2	8.06026E-06
	3	1.18005E-05
NiTiN	1	-6.7976E-05
	2	-6.7896E-05
	3	-6.4553E-05
NiTiN/T2	1	-7.8279E-05
	2	-8.4161E-05
	3	-7.4684E-05
NiWC	1	1.73633E-05
	2	1.70104E-05
	3	1.69712E-05
NiWC2	1	-1.5474E-05
	2	-2.4423E-05
	3	-1.7007E-05
NiWC3	1	-5.2421E-06
	2	-5.6331E-06
	3	-5.5918E-06
NiWC/T2	1	1.57799E-05
	2	1.65825E-05
	3	1.67878E-05

The NiWC3 showed very good performance with essentially no weight change, no apparent nickel layer and no apparent attack on the surface. Further analysis with a Zigo interferometer was conducted to measure the surface roughness changes. Initial indications show very good corrosion resistance of the cermets tested with the best performing being the NiWC or the NiWC3. Further work is needed to understand where the nickel surface layer on the NiWC came from. These cermets seem to perform far superior to other cermets (ZrCW cermet) in oxidizing environments and also slightly better in the salt environment.

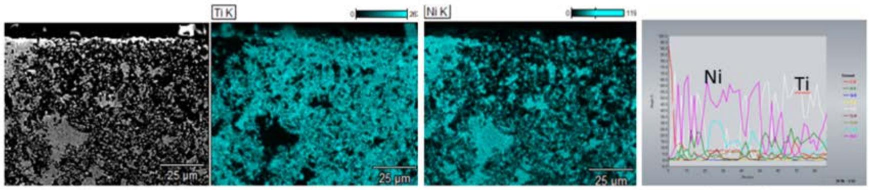
These tests demonstrated that the static immersions salt corrosion resistance of HybriMet is as good as or better than the control, Haynes 230.

Summary of surface roughness changes of samples

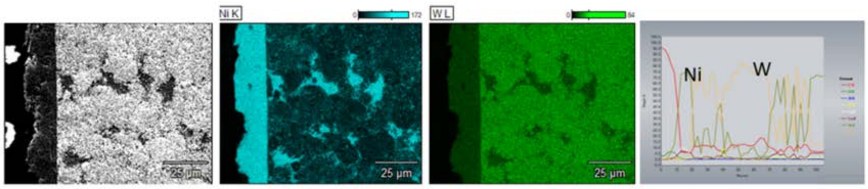
		Weight Change/Surface Area	
Sample Type	Sample #	Pre-Exposure RMS	Post-Exposure RMS
Haynes 230	1	1.07363E-05	0.826
NiTiN	1	0.198	2.774
NiTiN/T2	1	0.216	3.989
NiWC	1	0.504	1.324
NiWC2	1	0.514	1.324
NiWC3	1	0.440	0.320
NiWC/T2	1	0.498	1.293



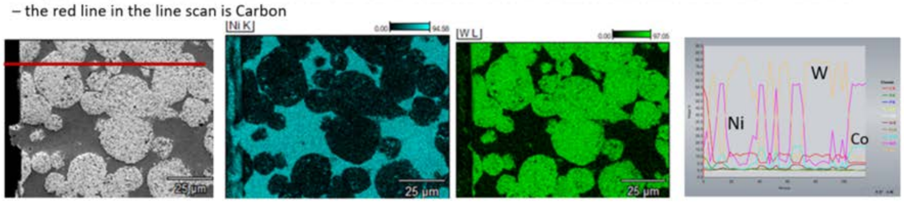
Haynes 230 sample exposed to MgCL:KCL at 750C for 250 hours. ~10-20um chromium depletion on the surface is seen (see line scan and EDS map). With some increased Cr migrating through the grain boundaries.



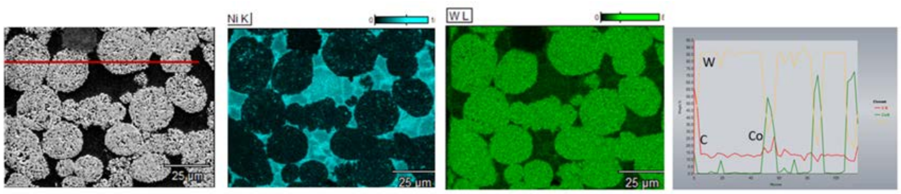
NiTIN sample exposed to MgCl:KCL at 750C for 250 Hours. Some porosity in the samples are seen along with some possible slight depletion of the of Ti on the surface. The Ni and TI is mixed fairly randomly in the sample



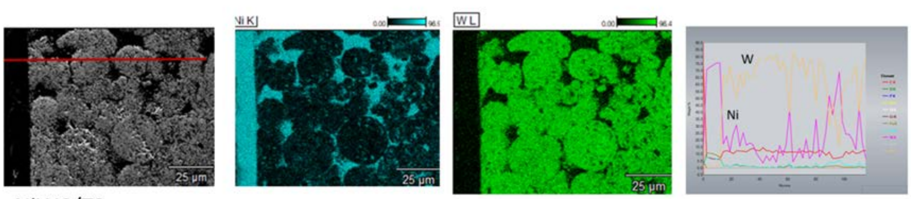
NiWC sample exposed to MgCl:KCL at 750C for 250 Hours. 10-15 um layer of nickel formed on the surface of the sample this layer seems to be well attached to the cermet surface once in the base cermet there is a mix of Ni and WC – the red line in the line scan is Carbon



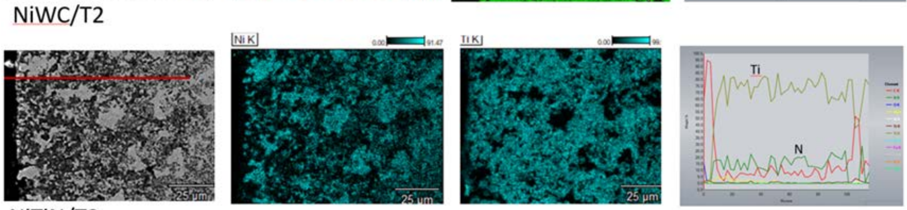
NiWC3 sample exposed to MgCl:KCL at 750C for 250 Hours. Very little attack and particles look to be intact and stable to the salt. The Nickel and Tungsten Carbide are clearly visible.



NiWC2



NiWC/T2



NiTiN/T2

Task 3. Erosion and Wear Test in Molten Salt

We modified a salt bath at the University of Wisconsin to incorporate a pin to contact a rotating disk to provide a measurement of the coefficient of friction and wear rate of subject materials. The friction is being measured by the load on the motor, the wear rate by profile and photographic analysis of the specimen after test and the erosion/corrosion by microscopic analysis. The wear test is essentially an evolution of the standard pin-on-disk tribological test. The current concept is shown in the figure on the right. In Phase II, we expect to exercise this equipment to develop a data base of candidate materials.

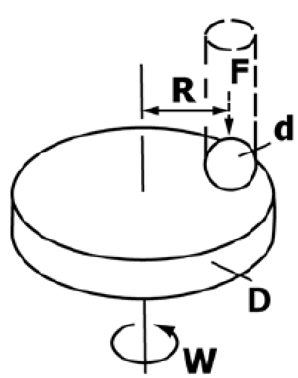
The specific objectives were to determine the relative rate of wear in molten salt of HybriMet NiWC and Haynes 230 and to develop bench wear test equipment and methodology for bearing materials in molten salt.

Pin on disk tests were conducted according to the ASTM G99-17 "Standard Test Method for wear testing with a Pin-on-Disk Apparatus"

The amount of wear in any system will, in general, depend upon the number of system factors such as the applied load, machine characteristics, sliding speed, sliding distance, the environment, and the material properties. The value of any wear test method lies in predicting the relative ranking of material combinations. In this pin-on-disk testing it is not possible to duplicate all the conditions that may be experienced in service, however we will attempt to reproduce the temperature and the corrosion environment. The load and, pressure, contact geometry, removal of wear debris may not be prototypic. However by conducting the ASTM standard test in with different material combinations at temperature in the salt the major parameters are captured and determination of the best performing material combinations can be determined. As these tests will be conducted according to Method 2 (under conditions not consistent with ILS data) the results will not be compared to data, however the parameters of the tests will be consistent between the tested materials and the test parameters/materials will be clearly documented.

Test Specimens:

In accordance with section 6.2 of the ASTM standard cylindrical pin with a spherical end with a diameter of 10mm and a disk in the range of 30-100 mm with thickness of 2-10mm were used in this study.



Apparatus:

The current pin on disk testing apparatus was custom designed and built for testing wear and friction rates between two metals, at a maximum temperature of 750C, in molten chloride salt, while maintaining a pure inert cover gas over the salt via a hermetically sealed test vessel. The basic function of the apparatus involves spinning a disc (submersed in molten salt) while a weighted pin rests on the disc.

Instrument:

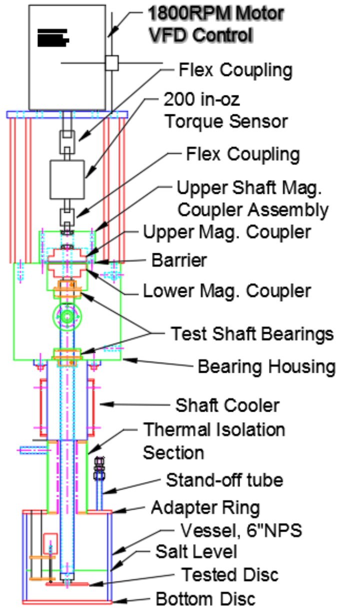


Figure 2: ACAD of Apparatus w/ Call-outs

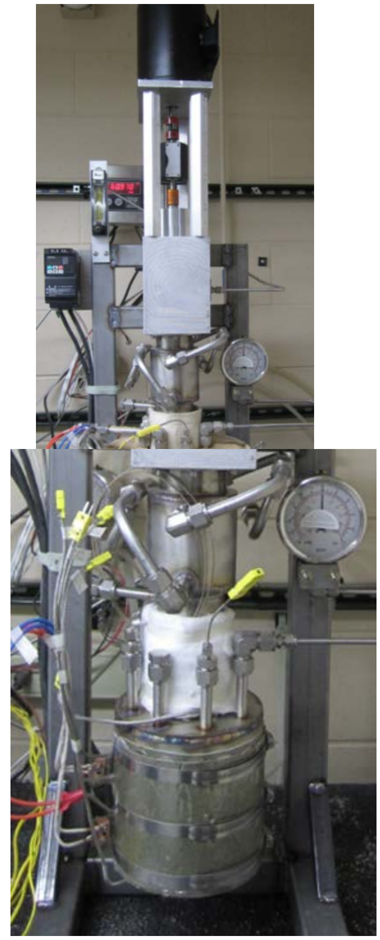


Figure 3: Test Vessel w/ Exposed Heaters and Wiring

The driving torque for the apparatus is provided by a top mounted 1800RPM, 1/4hp, 3 phase electric motor which is powered and speed controlled by a variable frequency drive. This motor directly drives the upper end of an Omega model TQ513-022 rotary torque sensor through a spider type flex coupling. The lower end of the torque sensor is flex coupled to the upper (outer) disc type magnetic shaft coupler assembly. A 1/16" thick, 316/L alloy disc magnetic barrier is sealed to the shaft bearing housing via a silicone o-ring, providing the hermetic seal between the test vessel and atmosphere. Below the magnetic barrier, the lower (inner) magnetic coupler is directly attached to the test shaft. The test shaft is fixed axially by bearings located at the top and bottom of the bearing housing. A shaft cooler is located directly below the bearing housing. Heat is removed from the test shaft via gas conduction across a small annular gap between the shaft OD and ID of the cooler body. This energy is ultimately removed by process chilled water flowing through an annular water jacket located on the OD of the cooler body. A thermal isolation section is located between the bottom of the shaft cooler section (20ºC), and the top of the test vessel (750ºC). The isolation section consists of two thin wall coaxial tubes welded together with rings on each end. The annular space between the tubes is filled with pourable high temperature insulation to minimize thermal conduction.

The test vessel is located directly below the thermal isolation section. A ¼" thick 316/L alloy adapter ring is welded directly onto the bottom of the thermal isolation section. The vessel wall consists of 316/L alloy 6"NPS pipe section, nominally 6.5" long. This pipe is directly welded to both the adapter ring above and a flat ¼" 316/L alloy disc below. All access ports into the vessel are located on the adapter ring and consist of thermal stand-off tubes welded to the adapter ring with a Swagelok compression fitting on the cold end of the tube, sealing and adapting to the required size.

Heat to the vessel was provided via a single resistive Inconel 600 serpentine heater wrapped around the OD of the test vessel, electrically isolated by phlogophite mica sheet. A second heat zone consisted of a high temperature Amtek heater tape wrapped around the OD of the lower end of the thermal isolation section. Both heaters were controlled via a National Instruments Labview PID routine, switched via solid state relays, and instrumented with multiple k-type thermocouples. One sheathed k-type thermocouple was inserted inside the test vessel, with the measuring point submersed in the salt near the spinning disc. This thermocouple was used to provide the test temperature.

Figure 4: Pin and Weight Loaded on Disc

Operation:

Operation was initiated by installing the test disc to the bottom of the shaft (internally threaded) via a ¼-20 bolt with a Belleville washer to maintain bolt tension at elevated temperature. After the disc was secured the pin and weight were installed, resting on the disc. With test articles installed, the vessel pipe section (with lower disc, heaters and external thermocouples already installed), was welded to the adapter ring. The vessel was then placed in an oversized insulation containment, and all ports were sealed. Pourable high temperature insulation was filled in around the vessel and all wiring was connected. The vessel was then evacuated four times, and filled after each evacuation with UHP Argon. Once flushing was complete UHP argon steady state flow was initiated, with the gas supplied at a port between the test shaft bearings, and exhausted through a test port at the adapter ring, at a flow rate of approximately 50 ml/min. Once flow rate was uniform, the heating for both a separate salt reservoir and the testing apparatus was energized.

When both the salt reservoir and testing apparatus attained a temperature of 550ºC, a salt transfer between vessels was completed, to a level of salt 1" above the top of the test disc. Once transfer was completed, test vessel temperature was ramped to the desired testing temperature. When the test temperature was attained, the motor was activated to the desired rotational speed, while temperature, torque and speed data was recorded until test completion.

Figure 5: Vessel (Red) exposed During Cool Down, 700C

When the test was complete, the test vessel temperature was reduced to 550ºC, and the salt was returned to the storage vessel via a dip tube. This is accomplished by pressurizing the test vessel with argon gas to which forces the salt from the test vessel to the storage vessel. When empty of salt, the test vessel was cooled to room temperature and the insulation, heaters, and wiring were removed. Once clear of obstructions, the 6" NPS vessel upper weld was removed, exposing the sample disk and pin. The disk and pin were then soaked in water to both facilitate release and remove any residual frozen salt. The pin, disc, and weight were then removed from the apparatus. Analysis of the samples were then conducted as indicated below.

Future Improvements:

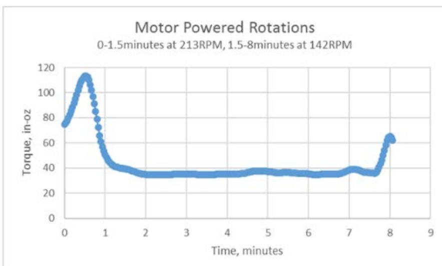
Operation of this test apparatus was sufficient to acquire initial data, but some future improvements may be required to attain high accuracy data in a reasonable amount of time. The motor for this design iteration was chosen for the ability to do testing at high rotational speed. It does not appear this high speed operation will be required, so a lower speed/higher torque motor should be acquired and installed for future testing. The torque sensor for this design was chosen mainly on short term availability. An alternative torque sensor should be acquired with both a higher allowable torque before failure and a higher accuracy signal. With the current design, the time required to prepare system for testing is extremely high. For future work, the design of the lower vessel should be modified or perhaps operated in a glove box to reduce cycle time.

Procedures.

1. Immediately prior to testing, and prior to measuring or weighing, the specimens were cleaned and dried with care taken to remove all dirt and foreign matter from the specimens.
2. Measure appropriate specimen dimensions to the nearest 2.5 μm or weigh the specimens to the nearest 0.0001 g.
3. Insert the disk securely in the holding device so that the disk is fixed perpendicular (+/-1°) to the axis of the rotation.
4. Insert the pin specimen securely in its holder and, if necessary, adjust so that the specimen is perpendicular (+/-1°) to the disk surface when in contact, in order to maintain the necessary contact conditions.
5. Add the proper mass to the system to develop the selected force pressing the pin against the disk.
6. Start the motor and adjust the speed to the desired value.
7. Set the revolution counter (or equivalent) to the desired number of revolutions.
8. Begin the test with the specimens in contact under load. The test is stopped when the desired number of revolutions is achieved.
9. Remove the specimens and clean off any loose wear debris. Note the existence of features on or near the wear scar such as: protrusions, displaced metal, discoloration, microcracking, or spotting.
10. Re-measure the specimen dimensions to the nearest 2.5 μm or reweigh the specimens to the nearest 0.0001 g, as appropriate.
11. Repeat the test with additional specimens

Initial Pin on disk Testing:

Initial testing was conducted on two pin on disc combinations, namely a NiWC disc vs. NiWC pin, and a Haynes 230 disc vs NiWC pin. Both tests were conducted at 725C, immersed in purified /reduced MgCl:KCL, and operated under an inert cover gas. Both tests were conducted with a 3.2mm(0.125in) diameter pin with a centered 10mm diameter ground onto the sliding end of the pin. Both pins had an approximate mass of 6.4g and both were weighted with the same block of mass 151.5g (total mass of pin on disc=158g:5.57oz, Pin load=1.5N). For both tests, the distance between the center of shaft rotation to the pin was 1.25in(3.18cm).



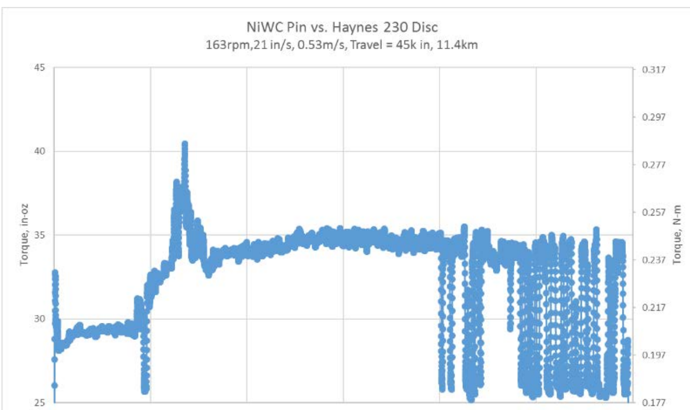
Torque Values fir NiWC vs. NiWC

Wear Track on(NiWC Pin vs). NiWC Disc

Figure 2Tip of NiWC Pin (vs NiWC Disc)

The initial testing utilized identical NiWC material for both pin and disc. Dry/cold testing indicated the instrument itself required 19.86 in-oz to operate without the pin on disc , while a cold/dry pin on disc total torque was 21.25 in-oz. This method attributed 1.39in-oz of torque due to the pin, indicating a coefficient of friction between pin and disc of 0.2, agreeing with previous data at room temperature. When the drive motor for the hot/wetted testing was energized, it was determined the testing system did not have adequate torque to reliably spin the disc. Total pin travel during test was 398m at approximate average velocity of 0.5m/s. Torque attributed to the pin varied between 3 and 96in-oz. Upon inspection the pin appears to have a metallic build-up on the tip while the disc appears to have possibly lost some material in the pin track (this is thought to be a nickel layer).

The second test utilized a Haynes 230 disc and NiWC pin.



Torque Values for NiWc Pin vs 230 Disc

Wear track on 230 Disc(vs NiWC Pin)

Tip of NiWC Pin (vs 230 Disc)

Dry/cold testing indicated a pin on disc induced torque of 2.56 in-oz, giving a coefficient of friction between these two materials of 0.37. The hot/wetted testing was operated over approximately 6 hours, at an average velocity of 0.53m/s, with a total travel of 11.5km. Pin attributed torque generally climbed during the 1st 1.5 hours of the test, from a level similar to cold dry testing to a peak of 16 in-oz. Pin attributed torque then decreased over 15 minutes to 8 in-oz of torque and remained fairly steady for 2.5 hours. Pin attributed torque throughout the last two hours of this test varied between two basic levels, 0.4 in-oz and 8.2 in-oz, residing at one level for some minutes and then switching to the other level. Testing was ended due to desired amount of travel having been attained. Upon inspection the disc appears to have been relatively unaffected by the testing (discolored in the pin track but no trunching), the pin however appears to have a similar buildup to the 1st test and the radius of curvature decreased.

It is theorized that the corrosion layer observe in the static corrosion tests is soft and builds up on the pin. It is expected that the NiWC, that exhibited no corrosion layer will be the better bearing material. This will be confirmed in future testing.

Significantly, this is the first pin-on-disk tribometer capable of running in a high temperature molten salt environment. This capability will accelerate the selection of bearing materials for high temperature salt applications and advance the understanding of wear mechanisms in molten salt.

Task 4. Mechanical Property Tests

This task supports objectives 1 and 3. In this task, mechanical and physical properties important to the high temperature molten salt application were measured on NiTiN. A similar set of data was obtained under a corporate development contract and is also presented in this report. This testing was conducted at Orton Labs, leaders in the testing of hard materials, and in house at Powdermet. Powdermet personnel coordinated with Orton to organize and manage the task. Coefficient of thermal expansion and thermal conductivity are critical to the design of molten salt pumps. The thermal expansion is measured to 1000ºC and thermal conductivity to 500ºC. The mechanical properties are measured at room temperature. This will provide a first set of data for comparison to the data on candidate alloys. During Phase II the mechanical strength over temperature will need to be measured.

The following mechanical and thermal properties were measured.

a) Coefficient of Thermal Expansion ASTM E-228 Thermal Linear Analysis. Numerical and graphical results show the percent of linear thermal change (expansion or shrinkage) versus temperature using an Orton dilatometer. This test will be conducted in air, heating and cooling at 3ºC /minute from room temperature to 1000ºC in air.

b) Thermal Conductivity. ASTM E-1461 Thermal diffusivity from room temperature to 500ºC is measured by laser flash. Three samples of 0.08", 0.12", and 0.16" will be tested. Thermal conductivity is calculated from the thermal diffusivity, heat capacity, and density. ASTM E-1269 Specific heat capacity is measured by differential scanning calorimetry.

c) Density will be calculated from dry measurements on the molded billets after surface grinding to provide a smooth surface.

d) Hardness was be measured using a Rockwell hardness tester and micro indentation techniques. Hardness as measured on surfaces as molded and ground to 10 µm or better for measurement accuracy were compared.

e) Tensile ASTM E8, tensile testing of metals, determines important mechanical properties such as yield strength, ultimate tensile strength, elongation, and reduction of area. Five micro dog bone specimens will be tested.

f) Flexural, ASTM C-1161 Flexural strength at ambient temperature is determined in three-point or four-point bending on bars of rectangular cross-section. Testing of ten specimens per type/brand is recommended by ASTM.

g) Fracture Toughness, ASTM C-1421 Fracture toughness at ambient temperature is determined in three-point or four-point bending on chevron-notched bars of rectangular cross- section. Testing of four specimens per type/brand is recommended by ASTM.

h) Compressive, ASTM E-9 Data obtained from a compression test includes the yield strength, the yield point, Young's modulus, the stress-strain curve, and the compressive strength. Four specimens will be measured, 2 each 12 mm diameter x 10 mm and 5.2 mm diameter x 10 mm.

HybriMet™ Metal Matrix Composite

Typical Properties

Property	Method	Conditions	Units	Typical Value[1] NiWC	NiTiN
Density	ASTM C134	RT	g/cc	12.35	7.02
Hardness	ASTM E18	RT	GPa	14.7	13.7
Vickers Hardness			HV (300)	1496	1396
Rockwell Hardness	ASTM E18	RT	HRC	60	56
Tensile Strength	ASTM E8	RT	Mpa	944	1032
			Ksi	137	150
Tensile Elongation	ASTM E8	RT	%	0.25	0.42
Elastic Modulus	ASTM E8	RT	GPa	352	293
			$x10^6$ psi	51	42.5
Flex Strength	ASTM C1161	RT	Mpa	761	643
			Ksi	110	93
Compressive Strength	ASTM E9	RT	Mpa	1467	1391
			Ksi	213	202
Compressive Modulus	ASTM E9	RT	Gpa	352	269
			$x10^6$ psi	51	39
Bulk Fracture Toughness	ASTM C1421	RT	Mpa*m^1/2	29.5	19.3
Coefficient of Friction	pin-on-disk	RT, dry		0.44	0.57
Coefficient of Friction	pin-on-disk	RT, lubricated		0.14	0.14
Coefficient of Friction	pin-on-disk	RT, sea water		0.12	0.19
Wear Rate	pin-on-disk	RT, lubricated	mm^3/N-m	<< 10^{-12}	<< 10^{-12}
Rolling Contact Fatigue		RT, 2.5 Gpa	cycles	> $60x10^6$	NA
Thermal Conductivity	ASTM E1461	0 to 500°C	W/m-°C	30	21
			BTU-in/hr-f	208	144
Specific Heat Capacity	ASTM 1461	0 to 500°C	J/g-°C	0.23	0.55
Thermal Diffusivity	ASTM 1461	0 to 500°C	cm^2/sec	0.11	0.06
Electrical Conductivity				high	high
Thermal Expansion	ASTM E228	0 to 750°C	ppm/°C	7.97	10.67

1. This data has been generated on early prototype samples of HybriMet materials and is representative of the materials produced. This data is not to be considered a specification. Customers should evaluate the suitability of these materials for their specific use.

03-10-2018

Comparative Mechanical Properties

Properties	Units	52100 chrome steel	Ceramic Si_3N_4	Incoloy 800H	Haynes 230	NiWC	NiTiN
Density	g/cm^3	7.81	3.25	7.95	8.97	12.35	7.02
Coefficient of linear expansion	$10^{-6}/^oK$	11.9	2.5	14.4	12.7	7.97	10.67
Tensile strength	Mpa	<935	<525	700	838	944	1032
Young's Modulus	Gpa	200	304	196	201	352	293
Hardness	Hv_{10}/HRc	700/62	690/61		998/40	1496/60	1396/56
Fracture toughness	$MPa/m^{1/2}$	17-22	6.1			29.5	19.3
Heat resistance	^oC	472	>800	800	>1200	750	>1000
Corrosion resistance	--	low	high	high	high	high	high
Non-lubricated friction	--	high	low			0.44	0.57

The HybriMet cermets compare favorably with bearing steel, engineering ceramics and super alloys.

Task 5. Process Assessment and Cost Estimate

This task supports objective 1, 5 and 6.

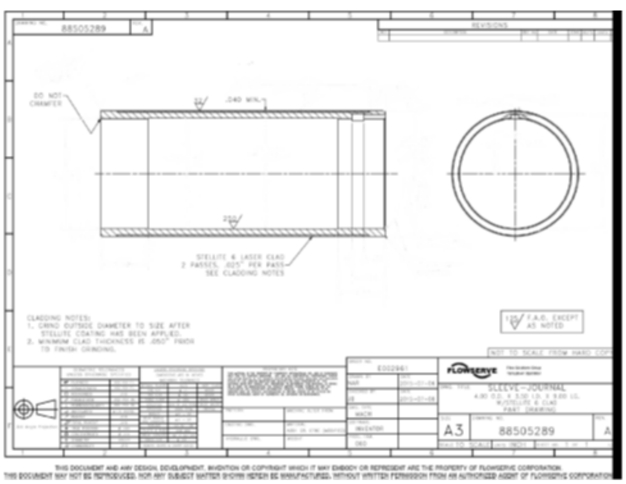


Fig 12. Typical Salt Pump Bearing -

Powder Production

Powdermet produced 4 grades of powders for consolidation; 3 NiWC grades and 1 NiTiN. The process of producing the powders is an evolution of a process to make flame spray coating material sold by Powdermet's spin out company MesoCoat. The powder grades used here are compounded for the ability to be consolidated into solid parts and to maintain the anti-corrosion properties of the flame spray grades.

Potential cost reduction ideas were reviewed. The flame spray materials have been in production for several years. It was determined that the highest potential for compounding cost reduction would be from increased production volumes. The process modifications to make the molding grade consist of simple, economical processes subsequent to the CVD process steps.

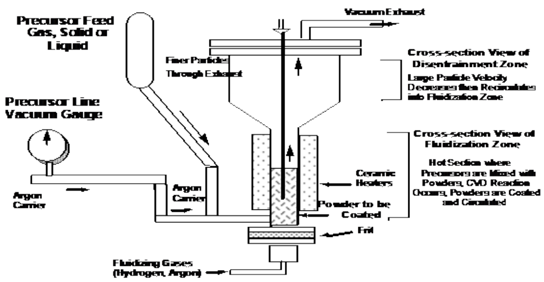


Figure 3 Benchtop CVD Reactor

Successful CVD, producing dense, adherent coatings, depends upon experimentally determining optimal deposition parameters. These parameters include the gaseous compound of the material to be deposited, substrate/powder temperature, gas concentration, flow, pressure and mixing within the reaction chamber, as well as coating thickness and substrate material. For the coating to have high integrity and substrate adhesion, the substrate must either have a similar coefficient of expansion to that of the deposited material or must form a strong chemical or metallurgical bond with it. The thinner the coating, the less similar the coefficients of expansion need to be. Where coating and substrate form no intermetallic bond and have widely differing coefficients of expansion, a good bond often can be achieved by using a thin interlayer of a third material.

Consolidation

Spark plasma sintering is capable of producing consolidated billets of varying diameters from <1/2in. up to over 1 ft. In this proof of concept program, Powdermet produced samples from 3 to 7 inches in diameter and ¼ to ¾ inches thick,

In spark plasma sintering, consolidation of the powder is brought about by the simultaneous application of pressure, vacuum and pulsed DC power. During SPS, the applied pulsed power generates micro plasma discharges between the adjoining powder particles that lead to neck formation and diffusion of matter between the particles and eventual closure of pores between the powder particles.

The control of this process will be key to maintaining the metal matrix nanocomposite properties and not alloying the interface coating into the metal matrix. The process variables were evaluated to predict the potential future process economics and to quantify process quality risks. We found that the molding conditions used produced dense billets in all 3 grades of NiWC, but allowed porosity in NiTiN. See the microphotographs on page 12 of this report.

To begin the process of evaluating the sensitivity of the metal matrix ceramic nanocomposite sintering temperature to corrosion resistance and physical properties, during molding billets for corrosion and mechanical properties test specimens a billet of NiWC and NiTiN were each held at sintering temperature for a period three times longer than specified in the current standard procedure. These materials were labeled NiWC/T2 and NiTiN/T2.

The hardness and density of NiWC/T2 are within one standard deviation of the hardness and density of the materials sintered using the current standard procedure. There is no significant improvement in hardness or density gained by the longer time at sintering temperature. The corrosion of NiWC is unchanged with the change in sintering conditions, but the initial results indicate that the corrosion of NiTiN sintered using the standard procedure is 18% less (better) than when held at sintering temperature for a longer time. NiTiN/T2 appears to be harder than NiTiN but there is a high variability in the NiTiN/T2 data because it was measured on a rough as-molded surface. Sintering time at temperature is important for some grades of HybriMet, but HybriMet NiWC has a reasonably broad processing window. There may be an opportunity for cost reduction by reduced time at temperature during molding.

		NiWC		NiWC/T2	NiTiN		NiTiN/T2
		average	std dev		average	std dev	
Hardness	HRC	59.5	0.52	60.4	56.2	0.49	58.7
Density	g/cc	12.36	0.228	12.57	7.02	0.05	7.05

Machining

Powdermet's nanocomposite is readily machined using EDM techniques and is ground using aluminum oxide media. All of the test specimens for the mechanical properties, corrosion testing were cut using wire EDM from molded billets. All of the EDM machining and grinding was completed at local shops. Machining rates are equivalent to those of super alloys. One significant difference is that these cermets do not have to be heat treated after machining to gain their high mechanical strength.

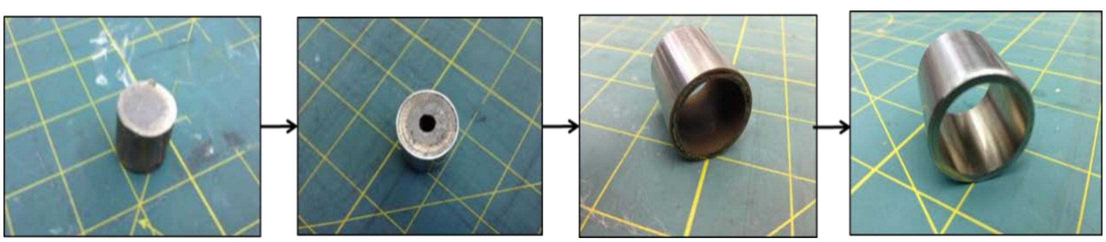


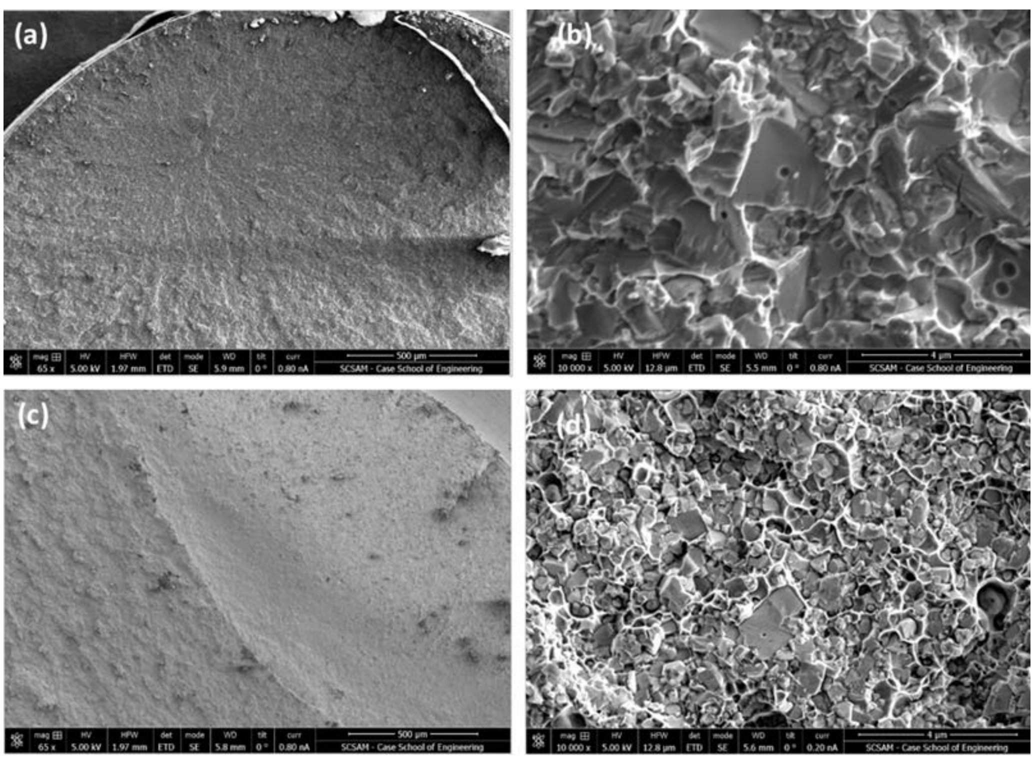
Figure 14 Lined sleeve bearing prototype production showing (from left to right) consolidated cermet, machined and inserted into bearing, EDM bored , white wheel ground/polished final part

Flaw Detection and Sensitivity.

Powdermet used microscopic analysis to assess the potential occurrence of flaws in the cermets consolidated by the processes studied and powder production methods. Microphotographs of NiTiN showed some porosity but showed NiWC to be well consolidated. See the photos on page 12 of this report.

Fracture surfaces of the mechanical test specimens were analyzed and showed well consolidated materials without flaws.

NiTiN **NiTiW**



Process Cost

Powdermet used a typical Flowserve salt pump bearing as an example to compare the cost of the part in Haynes 230 to the cost in cermet. The blank from which this bearing can be machined is a 4 inch tube with a ¼ inch wall, 9 inches long.

The cost comparison is very volume sensitive. Super alloys like Haynes 230 are produced in process that require high volumes. Powdermet's cermet powder is manufactured in large quantities and then molded to billets or shapes in a process that can economically produce low volumes.

Haynes International quoted a minimum order of a mill run of 1500 lbs of welded tube at $38,264.78 or $25.51/lb. For 10 bearings, likely a large quantity for this application, the cost of material would be $3,826/pc. Through a distributor, the cost of material for the bearing was quoted at $1689/ pc for enough material for 10 bearings. Chinese suppliers were as low as $20/kg for 100kg minimum orders, but small quantities ranges up to $1000/kg.

This same blank can be molded in cermet, at the current prototype pricing at about $200/kg or $1100/blank in a quantity of 4 pieces. This is 35% less that the 10 piece blank price from the distributor.

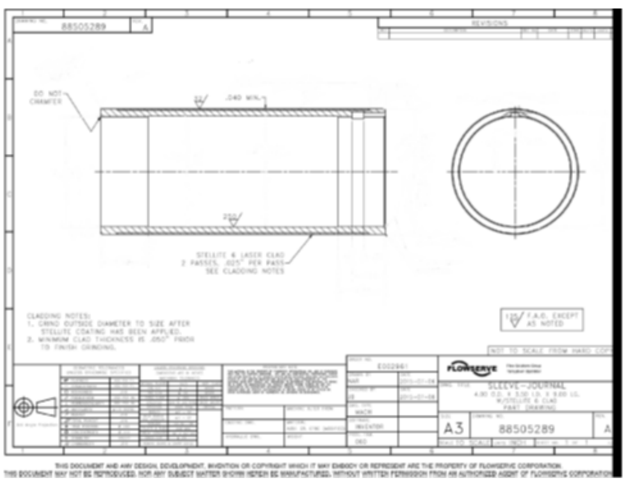


Fig 12. Typical Salt Pump Bearing -

The cost of machining of parts in HybriMet varies from equivalent to lower than that of super alloy billets. Heat treatment is a significant cost required for most super alloys to achieve advertised properties. Some alloys require machining, then heat treating and then a final grinding to attain finished tolerances.

During this project, molded billet size was increased by a factor of 9, demonstrating the production of more pounds per hour of machine time and the ability to produce large parts required in this CSP pump program.

HybriMet is more economical than many super alloys and a robust process promises a high production yield.

Task 6. Commercialization Plan

This task supports objectives 4 and 6. During this project, Powdermet began to build working relationships with molten salt pump suppliers and the system supply chain.

Fig 6. 0.015 HVOF HybriMet 360º bend without any spallation or cracking.

We found that most pump manufacturers are interested in our material for bearings seals and impellers, but none believe that other components of their pumps would survive in 750ºC chloride salts. Without a solution for the other pump components, there would be no market for HybriMet bearing and seals in CSP Gen 3 pumps. Powdermet therefore has expanded the HybriMet product to include a HVOF grade to coat other pump components. Since we are putting the corrosion resistance duty on the coating rather than the substrate, we have the opportunity to cost reduce the substrate and to engineer the thermal expansion match and adhesion chemistries.

Sulzer Pump has agreed to collaborate with Powdermet to manufacture a laboratory salt loop pump using Powdermet's HybriMet materials and to provide a quotation for a pump for the CSP Gen 3 demonstration. Sulzer has reviewed the data generated in this project and will evaluate HybriMet for another application in a subsea oil field pump.

IMPACT

Success in this project eliminates a barrier in the path to realize cost competitive solar power that can be dispatched 24/7. This product is based upon the successful anti-corrosion coatings developed by Powdermet. We believe that the product has a high probability of commercial success.

Pump manufacturers are reluctant to take on the risk and challenge of the technically difficult and distant ROI task to design and build a pump for 750ºC chloride salts. HybriMet metal matrix composites provide an economical solution for CSP as well as for industrial and commercial pump applications (such as product lubricated down-hole pumps).

This project:

1) Introduces a new class of materials, zero contiguity metal matrix ceramic composites, as a choice for high temperature molten salt pumps, particularly in KCL-MgCl2 salts over 750ºC.
2) Demonstrates a material that can be tailored for chemical resistance and coefficient of expansion without significantly degrading the mechanical strength.
3) Demonstrates a monolithic wear, erosion and corrosion resistant material that is not dependent upon a coating and limited in life by the coating thickness.
4) Establishes a methodology to generate wear data on bearing materials in molten salt and begin a data base on the material wear properties.

5) Provides the future potential of fabrication of parts beyond bearings through materials designed for improved formability and products for flame spray coating.
6) Delivers a pump for CSP Gen 3.

It has become apparent that to provide a useful pump solution, we need to expand the project beyond the molded components offered in this Phase I. For pump manufacturers to be able to quote and supply a CSP Gen 3 demonstration pump, they need a solution for all of the pump components. In Phase II and CSP Gen 3, we propose to develop a process to flame spray HybriMet cermet to coat pump shafts, housings and other components, providing a whole pump solution.

HybriMet is a platform technology, achieving mechanical properties that have been the goal, but out of reach, for the ceramics and super alloy industries for years. HybriMet is a novel ceramic reinforced metal composite that rivals super alloys. Measurements show NiWC toughness is 1.5 times higher than 52100 chrome bearing steel (29.5 $MPa/m^{1/2}$) and 5 times higher than Silicon Nitride and compressive strength 2 times higher than Silicon Nitride (1467 MPa). Tests demonstrated these nanocomposite materials offer very low friction when tested with a minimal amount of poorly lubricating fluids, and are extremely erosion and wear resistant, similar to diamond like carbon and Chromium Nitride coatings. The 250 hour, 750ºC static immersion tests in the chloride salt heat transfer medium of CSP have verified corrosion resistance near that of pure Ni and better than the best super alloys. Because no heat treating is required, cost is estimated to be less than super alloys.

The development of HybriMet metal matrix composites enables a path to corrosion resistance not available through alloys. As we have learned in the field of polymer composites over the last several decades, through composite technology we can separate the duties of the matrix and the reinforcements to achieve unique results. Here in this CSP pump application, we can choose the metal matrix for its corrosion resistance and choose the ceramic reinforcement for its mechanical and structural properties, providing an optimized material for the application.

The equipment and methodology developed in this project to measure the wear of materials in molten salt will for the first time enable to efficient evaluation and selection of materials for pumps in CSP applications. This equipment will be available at the University of Wisconsin Salt Corrosion laboratories.

This project will make CSP cost effective, and therefore solar power, cost competitive with fossil fuel derived energy for the first time. CSP can become a technology that can thrive and grow because of its economic value and not require government subsidies. With growth, the job and educational opportunities will proliferate.

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