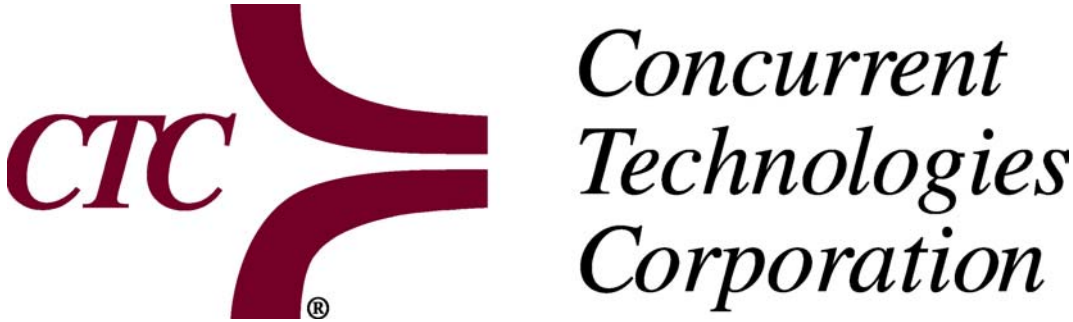

Evaluation of Acid Mine Drainage Residual Materials for Metallurgical Applications In Support of the Resource Recovery Initiative

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August 18, 2005
Mine Water Treatment Technology Conference
Pittsburgh, PA

Acknowledgements

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- *CTC* Technical Lead — Juan Valencia, *CTC*
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- Powder Metals Participants — Jim Aiello, St. Mary's Pressed Metals; Art Aiello, St. Mary's Pressed Metals

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Project Background

- **Primary Objective**
  - Evaluate specimens from AMD residual materials generated from various processes, which may recover valuable mineral resources, for the potential use as self lubricating bearing materials through metallurgical testing. In addition, make further recommendations for other viable applications.
- **Technical Approach**
  - Phase I – Materials Characterization of 8 residuals
  - Phase II – Powder Consolidation and Mechanical Testing for selected residuals
  - Summary Report
- **Primary Output**
  - A Summary Report containing all analytical data and recommendations for viable applications for each AMD resource recovery specimen evaluated.

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8 AMD Residual Specimens

Sample Number	Vendor	Resource Recovery Process
1	DTIC	DTIC Process
2	LTV Treatment - Banning (supplied by Fraunhoefer)	Conventional lime treatment with aeration and polymer
3	LTV Treatment - Clyde (supplied by Fraunhoefer)	Conventional lime treatment with aeration, polymer and sludge recirculation
4	Virotec	ViroMine® Process: oxidize with peroxide; neutralize the acidity with a combination of Bauxsol®, dolomitic lime and magnesium oxide and remove the metals with Bauxol®
5	Brandy Camp Treatment	Conventional treatment with aeration and polymer; separated with a belt filter press
6	Little Toby Creek	Limestone diversion wells; separated in sedimentation ponds
7	Damariscotta	Limestone diversion to precipitate the aluminum product
8	Iron Oxide Technologies, LLC	AIS/SBR Treatment (Activated Iron Sludge/Sequenching Batch Reactor)

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Phase I – Metallurgical Characterization

- Dry the following 8 specimens to remove all moisture
  - Topper Run
  - LTV-Banning
  - LTV-Clyde
  - Virotec
  - Brandy Camp
  - Toby Creek
  - Darmariscotta
  - Iron Oxide Technologies
- Receive chemistry compositions from Fraunhofer and/or the respective vendor
- Perform XRD (X-Ray Diffraction) analyses
  - Phase and crystal structure identification
- Perform SEM (Scanning Electron Microscopy) analyses
  - Particle size and distribution
- Perform LOM (Light Optical Microscopy)
  - Phase distribution and morphology
- Evaluate data for potential metallurgical applications

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Phase I – Conclusions

- Observed that all residuals, excluding the Darmariscotta AMD material, were very rich in iron oxides.
- Detected high concentration of calcium oxide and carbonates in the AMD residual materials after neutralizing processes.
- Determined AMD residual materials may not be suitable for producing structural components without a further treatment to eliminate all volatile components.
- Conducted XRD and found that most of the iron oxides are amorphous materials and the content of crystalline phases in the AMD materials is relatively low.
- Performed Optical and SEM analyses and found that AMD residual materials are made from clusters of primarily iron oxide sub-micron particles.
- Observed that AMD clusters are relatively homogeneous and have a small proportion of crystalline phases uniformly distributed.

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Phase II – Mechanical Testing

- Select optimum powder blends based on the previous work conducted by St. Mary's Pressed Metals
- Mix and perform green compaction of the selected powder-metal blends
- Consolidate and sinter powder mixture to form a metal matrix composite (MMC)
- Evaluate the microstructure of each MMC
  - Optical and SEM evaluations
- Mechanical Testing
  - Prepare specimens
  - Analyze for tensile and compressive strengths
  - Analyze for hardness
  - Analyze for wear

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Phase II – Powder Blends

AMD Residual	Bronze/Residual Ratio, weight %	Mixing by Attrition Milling	Acrawax Binder, weight %	Turbula Mixing, minutes
Brandy Camp	80/20	• 100 RPM • 30 minutes	0.75	
Brandy Camp	80/20		0.75	30
Iron Oxide Technologies	80/20		0.75	30
Bronze	100		0.75	30

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Example of a Green Compact Composite

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Green Compaction Results

Material	Sample Set ID	Mixing Method	Average Sample Weight, grams	Average Sample Density, g/cm^3
Bronze	BZ	None	31.60 ± 0.15	7.44 ± 0.006
Bronze/ Brandy Camp	BZ/BD/A	Attritor	21.10 ± 0.12	5.20 ± 0.017
Bronze/ Brandy Camp	BZ/BD/T	Turbula	20.43 ± 0.19	5.07 ± 0.10
Bronze/ Iron Oxide Technologies	BZ/IOT/A	Attritor	22.38 ± 0.014	5.43 ± 0.03

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Mechanical Testing Results

Material	Total Sintering Time, hours	Sample Density, g/cm^3	Approximate Compressive Yield Strength 0.2%,ksi	Ultimate Strength at Flaw Initiation, ksi	Approximate Strain at Ultimate Load, %
Bronze CT1000*	-	**7.2**	**22.0**	-	-
Bronze/ Brandy Camp - A	0.5	4.88	5.3	20.1	22.9
	1.0	4.92	5.5	21.0	24.2
	4.5	4.90	6.1	22.5	26.2
	5.0	4.96	5.3	22.8	25.9
Bronze/ Brandy Camp - T	0.5	4.80	6.2	28.1	30.7
	1.0	4.85	6.9	31.1	33.0
	4.5	4.86	6.3	33.7	35.8
	5.0	4.89	6.5	33.2	34.9
Bronze/ Iron Oxide Technologies - A	0.5	6.80	24.8	71.6	23.7
	1.0	6.92	24.2	76.0	25.4
	4.5	7.04	24.8	80.5	25.7
	5.0	7.01	23.6	79.4	25.2

A = Attritor Mixing
T = Turbula Mixing

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Mechanical Testing Results - continued

Sample ID	Hardness Vickers	Mass Loss, mg	Mass Loss, %	Initial Sample Roughness, µm	Final Sample Roughness, µm	Weight Ring Loss, mg	Scar Width, mm	Scar Block Volume, mm^3
Bronze/Brandy Camp - A	79.8	9.45	0.17303	3.19	3.64	-0.29	2.870	0.717
Bronze/Brandy Camp - T	N/M	53.43	0.97454	3.85	1.70	6.02	6.888	10.002
Bronze/Iron Oxide Technologies - A	86.8	18.04	0.24034	2.20	1.58	15.41	4.227	2.295

A = Attritor Mixing
T = Turbula Mixing

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Phase II – Conclusions

- Noted that 2 Bronze/Brandy Camp and one Bronze/Iron Oxide Technologies AMD mixtures had a very good green compactability.
- Achieved higher density for the bronze/Brandy Camp mixture using the mixture produced in the attritor mill.
- Observed that all composite mixtures exhibited weight loss during sintering at 850°C.
  - Bronze/Brandy Camp composite demonstrated the largest weight loss.
- Detected density changes in AMD residual materials after sintering.
  - Bronze/Brandy Camp composite experienced density decrease. This may be attributed to the decomposition of the carbonates and chemically-bonded water within the AMD residual particles.
  - Bronze/Iron Oxide Technologies composite had a substantial increase in density.
- Performed Optical and SEM microstructural analysis and observed uniform microstructure in the bronze composites.

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Phase II – Conclusions Cont.

- Discovered extensive particle pullout in the Bronze/Brandy Camp composite, which is an indication of poor bonding among the AMD residual particles.

- Detected that AMD materials have compressed yield strengths different than that of pure bronze.
  - The Bronze/Brandy Camp composite exhibited poor compression properties and a much lower yield strength than pure bronze.
  - The compressive yield strength of Bronze/Iron Technologies composite material was 11% and 30% higher than that of the Bronze and Bronze/Brandy Camp composites, respectively.

- Calculated that the maximum or ultimate strength of failure (peak stress at which the fracture of the specimen occurred) of the Bronze/Iron Oxide Technologies composite is more than twice the failure strength of the Bronze/Brandy Camp composites.

- Conducted wear resistance tests on AMD materials.
  - The Bronze/Brandy Camp composite material mixed in the attritor mill had the best wear resistance of all three materials.
  - The Bronze/Iron Oxide Technologies composite caused the most wear of the steal ring.

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Recommendations for Future Work

- Conduct thermogravimetry analyses (DTA/TGA) and DSC (differential scanning calorimetry) followed by XRD and XRF (X-ray fluorescence) spectroscopy to understand the phase evolution in the AMD materials.
- Investigate drying and milling the AMD residual materials, and conduct green compaction and sintering studies to try to understand the materials' ability to be compacted into solid products.
- If the metal matrix composites (MMCs) for increased wear resistance can be produced, select the most stable and chemically uniform AMD material to study.
- Identify the optimum volume fraction of AMD material that could be incorporated in the MMC without degrading mechanical properties.
- Analyze several MMC systems including stainless steels, aluminum alloys, and copper alloys.
- Study the mixing and sintering ability of those high iron oxide content AMD residuals with other materials such as magnetite to produce permanent magnets.

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Potential Applications for the Various AMD Residual Materials

AMD Residual	XRD		Potential Applications
Topper Run	Amorphous iron phases	Major	• Magnets using ferrous matrix composites with magnetic properties • Additive for cement and other construction and ornamental materials
	Quartz SiO_2, Calcite $CaCo_3$	Minor	
	Dolomite $CaMg(CO_3)_2$	Minor	
	Bassanite $CaSO_4 \cdot ½H_2O$	Minor	
	Rutile TiO_2	Minor	
LTV-Banning	Amorphous iron phases	Major	• Pigments for paint, wood stain plastic. (Milled to submicron size) • Pigment for cement, brick, tiles
	Calcite $CaCO_3$	Minor	
LTV-Clyde	Amorphous iron phases	Major	• Pigments for paint, wood stain plastic. (Milled to submicron size) • Pigment for cement, brick, tiles
	Calcite $CaCO_3$	Minor	
Brandy Camp	Amorphous iron phases	Major	• Additive for cement and other construction and ornamental materials
	Calcite $CaCO_3$	Minor	

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Potential Applications for the Various AMD Residual Materials

AMD Residual	XRD		Potential Applications
Virotec	Amorphous iron phases	Major	• Magnets using ferrous matrix composites with magnetic properties • Additive for cement and other construction and ornamental materials • Pigments for paint, wood stain plastic. (Milled to submicron size) • Pigment for cement, brick, tiles • Ceramics, glass
	Hermite Fe_2O_3	Minor	
	Quartz SiO_2	Minor	
	Calcite $CaCO_3$	Minor	
	Anatase TiO_2	Minor	
	Gibbsite $Al(OH)_3$	Minor	
	Boehmite AlO(OH)	Minor	
	Sodalite	Minor	
Toby Creek	Amorphous iron phases	Major	• Magnets using ferrous matrix composites with magnetic properties • Abrasive additives • Additive for cement and other construction and ornamental materials • Pigments for paint, wood stain plastic. (Milled to submicron size) • Pigment for cement, brick, tiles • Ceramics, glass
	Quartz SiO_2	Minor	
	Muscovite $KAl_2(Si_3Al)O_{10}(OH)_2$	Minor	
	Kaolinite $Al_2Si_2O_5(OH)_4$	Minor	

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Potential Applications for the Various AMD Residual Materials

AMD Residual	XRD		Potential Applications
Darmariscotta	Amorphous Al/S/O/Si/Na phase	Major	• Abrasive additives • Metal matrix composites • Cement additive
	Amorphous iron phases	Minor	
	Zinc sulfide ZnS	Minor	
Iron Oxide Technologies	Amorphous iron phases	Major	• Magnets using ferrous matrix composites with magnetic properties • Metal matrix composites • Additive for cement and other construction and ornamental materials • Pigments for paint, wood stain plastic. (Milled to submicron size) • Pigment for cement, brick, tiles • Ceramics, glass

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Summary Report

- A Summary Report was written and submitted to the SAC and PaDEP. The report include the following:
  - Compiled raw data (Phase I and II)
  - Characterization of the data from Phase I
  - Evaluation of the Phase II test results
  - Recommendations for future work in this area to investigate the true potential for the self-lubricating bearing application
  - A brief discussion on other potential metallurgical applications for all AMD residuals that may be further investigated
- A copy of this report can be obtained for your review.

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Back Up Slides on *CTC* Capabilities

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CTC Capabilities

- An independent, nonprofit, applied research and development professional services organization
- Provide management and technology-based solutions to a wide array of clients representing state and federal government as well as the private sector
- Core technical competencies
  - Environmental technologies and management
  - Chemical, mechanical and metallurgical engineering
  - Risk management
  - Life cycle cost analyses
- Working relationships in the powder metallurgy industry and understands the performance enhancements provided by source materials
- Fulfill project integration to transition technical results from the applied research and development phase to full-scale industrial operations to provide significant economic and environmental benefits

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CTC Metallurgical Capabilities

- Powder metallurgy laboratory
  - Powder characterization
  - Blender, presses, powder compaction dies, sintering furnace
- Materials characterization laboratory
  - Sample preparation equipment
  - Optical and SEM microscopes equipped with image analysis systems and Energy Dispersion Spectroscopy for micro chemistry analysis
  - Chemical analysis
- Materials testing laboratory
  - MTS and Tinius-Olsen testing machines for low and high temperature tension, compression and fracture toughness
  - Impact testing machines for room and cryogenic temperature testing
  - Powder compaction testing
  - Hardness and microhardness
  - Fatigue testing machines
- High Temperature thermophysical properties laboratory
  - Specific heat, thermal expansion, thermal diffusivity and conductivity

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