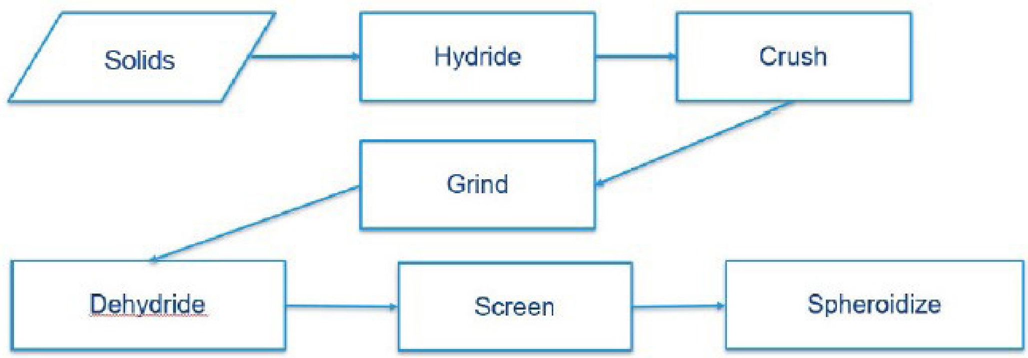
SUCCESS STORY 5535.001

Best practices result in documented impurity evolution of hydride-dehydride process

Oxygen levels of 500-700 ppm may be achievable if partical size distribution is increased



Conventional HDH process flow

PROBLEM

Most prior work focuses on spherical powder feedstock, but the use of angular powder for laser powder bed fusion (LPBF), cold spray, and electron beam melting is also actively researched. The use of spherical powder maximizes powder density and can exhibit improved flowability compared to angular powder. However, yields in the typical 15-50 µm particle size distribution are low, and the bar stock must be suitably processed before atomization on the large-scale Electrode Induction Melting Gas Atomization (EIGA) equipment. The hydride-dehydride (HDH) process of producing angular powder may allow for significant cost and lead time savings if powder with adequate density, flow, and chemistry can be achieved.

OBJECTIVE

The purpose of this study was to determine if the contamination level for niobium alloy powders processed via the HDH route, using laboratory-scale equipment and novel production practices, could achieve similar levels of contamination (e.g., <300 ppm oxygen) compared to powders processed using current state-of-the-art Electrode Inert Gas Atomization. This effort informed government and defense entities on the possibilities and limitations of the production of HDH niobium alloy powders with regards to contamination levels and viability of scale-up for use in additive manufacturing (AM) technologies requiring a particle size distribution of 15-50 µm.

AMERICA MAKES TECHNOLOGY DEVELOPMENT ROADMAP

This project aligns to:

MATERIAL

ASTM PROCESS CATEGORY
Powder Bed Fusion

EQUIPMENT
Electrode Induction Melting Gas Atomization (EIGA) Equipment

MATERIAL
Hydride-dehydride (HDH) Powders, Niobium Alloy C103

TECHNICAL APPROACH

Most of the proposed project was focused on identifying the processing steps that had the greatest impact on contamination pickup. Compositional analysis of the alloying and trace elements included Nb, Ta, Hf, Zr, W, Fe, Al, Ni, Co, V, Cr, and Cu and were measured after each processing step where there was an assessed potential for contamination. Oxygen, nitrogen, carbon, and hydrogen interstitials were of most interest and measured after every processing step. Oxygen can be difficult to measure when the material is in hydride form so appropriate uncertainty was communicated. The processing steps of interest included the initial C103 feedstock, hydriding, crushing, grinding, dehydriding, screening, and spheroidizing (optional).

ACCOMPLISHMENTS

The project demonstrated best practices that can be implemented in each processing step to reduce oxygen, nitrogen, hydrogen, and carbon impurity contamination. It was determined that current equipment can produce fine C103 angular powder with <1000 ppm oxygen, but a larger size fraction and investments in dehydriding and grinding equipment are required to reduce oxygen further to ~600 ppm oxygen. It was also evident that achieving 300 ppm oxygen levels does not appear possible for the 15–53-micron size range of interest. This higher oxygen content may impact the ability to use the powder in different additive technologies such as laser powder bed fusion, direct energy deposition, and cold spray. However, the levels achieved in this work may still produce adequate properties and this should be tested in future research.

PROJECT END DATE

September 2023

EXPECTED DELIVERABLES

- 20 lbs. of HDH C103 powder
- 4 lbs. of spheroidized HDH C103 powder
- Final report

FUNDING

$225,000 total project budget

PROJECT PARTICIPANTS

Project Principal:
ATI Materials

Other Project Participants:
NCDMM/America Makes

Public Participants:
U.S. Department of Defense