Fall 12-1-2012

Effect of High Energy Ball Milling on Transformation Temperature of Cr2Nb

Jackson Smith

Case Western Reserve University

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Department of Materials Science and Engineering, Case Western Reserve University, Cleveland, OH

ABSTRACT

The focus of this research was to determine a milling procedure to help facilitate the Cr_2Nb transformation. The metric to evaluate a facilitated phase transformation was a reduction in endothermic onset temperature during Differential Scanning Calorimetry (DSC). The transformation of the Cr+Nb powder to Cr_2Nb alloy is diffusion dependent, therefore by creating more intimate contact the elements will be closer together. A reduction in spatial distance for a diffusion-based reaction will lead to a reduction in total time for the reaction to complete. The design of a multi-stage milling process allows the refinement of the powder particle size so that diffusion can more easily occur at a lower temperature. The effectiveness in powder size reduction was tested for two bearing sizes to determine the required milling time for each stage. It was determined that dry milling elemental Cr powder for 5 minutes with 4.88mm bearings and then wet milling for 3 minutes with 2.47mm bearings produced a size distribution with a D90 less than 25μm. After determining this 2-step milling procedure, a mixture of Cr+Nb powder was milled with the 2-step process and compared with Cr+Nb powder that had been milled with a 1-step process. X-Ray Diffraction (XRD) was done on the mixtures before DSC and the results showed the presence of elemental Cr and Nb. These two mixtures were then exposed to a controlled heating rate of 20 degrees C per minute up to 1400C in a Netzsch Pegasus 404 F1 DSC and then cooled. Post-DSC, the XRD results showed a significant increase in the presence of Cr_2Nb alloy. Therefore, the endothermic reaction visible in the DSC results must have been the phase transformation from elemental Cr and Nb to Cr_2Nb alloy. In comparing the 1-step and 2-step milling processes, the DSC results show that the transition temperature of the Cr+Nb powder was reduced from 1229C to 1089C when the milling was changed from 1-step to 2-step. Future research will investigate the effects of such milling on the in-situ formation of Cr_2Nb during additive manufacturing processes.

INTRODUCTION

When elemental powders are in-situ alloyed during additive manufacturing one of the factors to be considered is the phase transformation temperature needed to get the desired alloy. Something that can affect the transition temperature is the chemical and morphological characteristics of the elemental powders used to create the part. By designing a multi-step milling procedure for the powder, it can be possible to reduce the size of the powder particles and increase contact between the elements. Reducing the particle size of the powders reduces the diffusion distance needed to obtain homogeneity and can improve the alloying reaction speed. The combination of these two effects means that it could be possible for powder refinement to aid in reducing the phase transformation temperature for in-situ alloying. In order to design a multi-step milling process the elemental powders must be milled using progressively smaller bearings. First, larger bearings are used, and the powder is milled in small time increments and imaged after each increment. Using the images of the powder, the size distribution can be determined for each milling time. By comparing the 10th, 50th, and 90th percentiles (D10, D50, and D90) of the powder diameter for each milling time increment, the milling time required to reach a minimum size for that bearing size can be determined. The same process can be repeated for smaller bearing sizes until the desired powder size distribution is reached. The milling time required to reach a minimum size at each bearing size can then be used to design a multi-stage milling process. For this experiment only two bearing sizes were used, 4.88mm and 2.47mm diameter, in order to reach a desired size distribution of a D90 less than 25μm.

MATERIALS AND METHODS

•Pure Cr powder and pure Nb powder were used. The Cr+Nb powder mixture was 15.785g. 8.4244g of Cr and 7.3606g of Nb making a 2.05:1 atomic ratio of Cr to Nb.

•A Spex Sample 8000 mixer/mill equipped with a hardened steel Spex 8001 sample chamber was used. Two sizes of stainless steel bearings were used, 4.88mm diameter and 2.47 mm diameter. For each bearing size, the pure Cr powder was milled for 1,2,3,4,5, and 10 minutes.

•For 4.88mm bearings: 15.88g of Cr powder was dry milled with the bearing to powder ratio by weight being 10:1.

•For 2.47mm bearings: 19.16g of Cr powder was wet milled with a 2:1 ratio by volume of powder to isopropyl alcohol. The bearing to powder ratio was also 10:1. The wet powder was dried under a halogen lamp.

•For the 1-step mixed power, Cr+Nb powder was milled for a substantial amount of time in a planetary mill at NASA GRC.

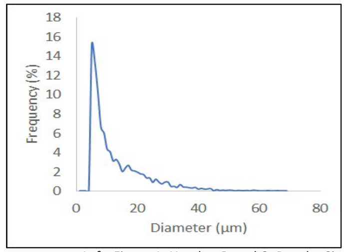
•For the 2-step mixed powder, Cr+Nb powder was dry milled for 5 minutes with the 4.88mm bearings and a 10:1 bearing to powder weight ratio then wet milled for 5 minutes with a 2:1 powder to acetone volume ratio. The wet powder was dried under a halogen lamp.

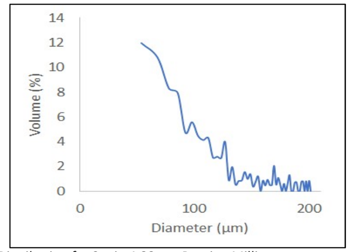
•Powder images were taken using a binocular microscope with the sample backlit on a plastic weight paper. All images taken were analyzed using an ImageJ macro to retrieve data on the size of the particles. The data obtained was imported into Excel and hand filtered.

•A Netzsch Pegasus 404 differential scanning calorimeter (DSC) was used to analyze the thermal transitions of the Cr+Nb powder. For the test, the sample was placed in a Y_2O_3 crucible with a lid and heated to 1400C, cooled to 400C, and then reheated up to 1400C.

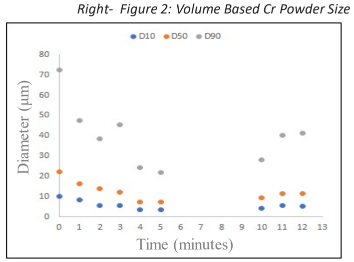
•A Bruker Discover D8 (1 dimensional XRD) with a Copper Kα radiation source was used. The sample was scanned from 15° to 90° 2θ resulting in a 30-minute scan. The information was interpreted with the native Bruker XRD software, DiffracEva.

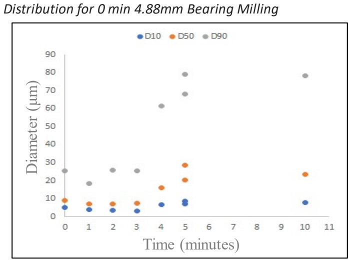
Figures





Left- Figure 1: Number Based Cr Powder Size Distribution for 0 min 4.88mm Bearing Milling
Right- Figure 2: Volume Based Cr Powder Size Distribution for 0 min 4.88mm Bearing Milling





Left- Figure 3: 4.88mm Bearing Powder Diameter Changes
Right- Figure 4: 2.47mm Bearing Powder Diameter Changes

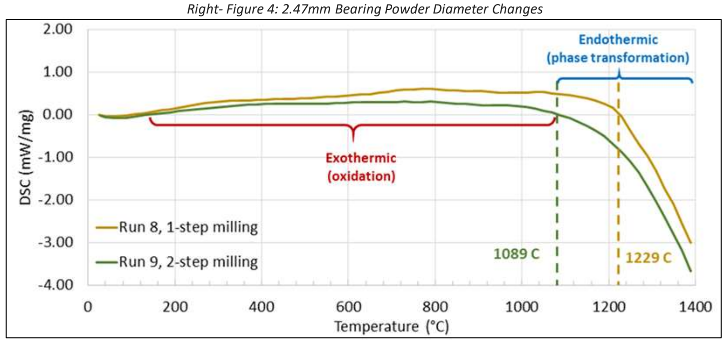
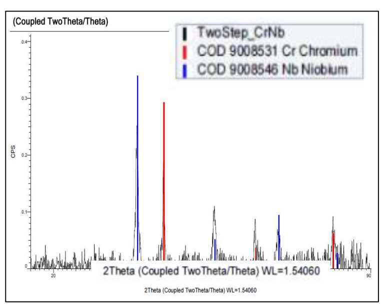
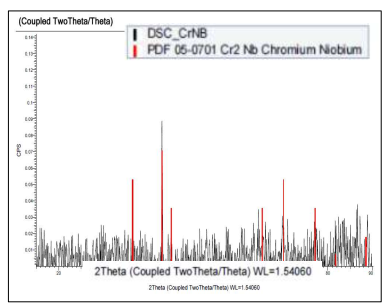


Figure 5: Summary of DSC Results





Left – Figure 6: XRD of Cr+Nb powder prior to DSC
Right – Figure 7: XRD of Cr+Nb powder after DSC

RESULTS

- In Figures 1 and 2 the original size distribution in frequency as well as volume percent can be seen for the pure Cr powder.
- From milling the pure Cr powder with 4.88mm bearings the minimum size of particles could be achieved by milling for 5 minutes. This minimum can be seen in Figure 3 where a D90 of 21.7μm was achieved.
- From milling the pure Cr powder with the 2.47mm bearings, 3 minutes was needed to reach the minimum size distribution. This minimum can be seen in Figure 4 where the D90 reaches a minimum of 25.6μm at 3 minutes.
- For the Cr and Nb mixed powder, a 1-step and a 2-step sample were created and compared so the effectiveness of the two-step process could be determined.
- The 2-step powder was analyzed using XRD before being put through the DSC. In Figure 6, two distinct peaks corresponding to elemental Cr and Nb can be seen.
- The 1-step and the 2-step powder were heated at a controlled heating rate of 20 degrees C per minute up to 1400C in the DSC in order to measure the phase transition temperature at which the powder transforms to Cr_2Nb. In both DSC runs some oxidation of the crucible occurred, however the endothermic reaction from the phase transition is still visible and can be seen in Figure 5. By using the 2-step milling procedure the phase transformation temperature was reduced from 1229C to 1089C.
- The XRD analysis from the post-DSC 2-step powder shows a significant increase in the presence of Cr_2Nb. This is indicated by a single peak corresponding to Cr_2Nb seen in Figure 7.
- The combination of the XRD and DSC results show that the endothermic reaction that occurred in the DSC corresponds to the phase transformation to Cr_2Nb and that this transformation temperature was reduced using the 2-step milling process.
- Overall, the data collected on the milling process permitted the creation of a multi-step milling process to facilitate transformation to Cr_2Nb. The powder refinement from the 2-step milling process helped to reduce the phase transformation temperature for Cr_2Nb.

CONCLUSIONS

- Minimum D90s of 21.7μm and 25.6μm were achieved with 5 minutes and 3 minutes of milling using the 4.77mm and 2.47mm bearings respectively.
- The Cr+Nb powder milled by 1-step and 2-step processes had phase transformation temperatures of 1229C and 1089C respectively, showing a 140 degree decrease attributed to the milling process.
- Pre-DSC XRD results show the mixed powder contained elemental Cr and Nb whereas the post-DSC XRD results show a great increase in Cr_2Nb, confirming the endothermic reaction exhibited in the DSC was the phase transformation to Cr_2Nb as well as the beneficial effects of the milling processes investigated.

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ACKNOWLEDGEMENTS

Support provided through SOURCE by the University and Case Alumni Association.
Thank you to Dr. John Lewandowski, Dr. Dave Ellis, and David Scannapieco for their help on this project as well as access to facilities in the Advanced Manufacturing and Mechanical Reliability Center (AMMRC)