JOURNAL OF
NATURAL SCIENCES AND MATHEMATICS RESEARCH

Available online at http://journal.walisongo.ac.id/index.php/jnsmr

The effect of Milling Time on Crystal size Sandvik Sanergy

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Received: 10 May 2020, Revised: 28 May 2020, Accepted: 23 June 2020.

Abstracts

Preliminary study material sandvik sanergy as an alternative material for interconnect applications solid oxide fuel cell (SOFC) have been conducted. Sandvik sanergy was milled for 0, 8, 24, and 48 hr with ball milling planetary MTI Corp. The physical properties of small grain size can affect the strength of the alloy when applied as interconnect. In this study, ratio between ball miller and sandvik sanergy was 1:10 aiming to obtain a small grain size. Crystal size characterized by XRD Rigaku Smartlab, morphology of sandvik sanergy after milling observed using SEM Hithaci SU3500 20 kV. Diameter of particles were observed by particle size analyzer (PSA). Results showed that crystal size as well as particle size tended to decrease with the increasing milling time. Discrepancy occurred at 24 hour of milling time and showed agglomeration.

Keywords: sadvik sanergy, crystal size, milling, morphology sandvik

1. Introduction

Interconnect materials have a very significant role in the performance of solid oxide fuel cells (SOFCs) which are currently being developed. The interconnect in one SOFC system functions as a liaison between cells. Each cell consists of an anode, a cathode and an electrolyte. The operating temperature for SOFC is generally higher than 1000^{o}C. Efforts to reduce the operating temperature to 700oC are able to make SOFCs an alternative to green energy change (Werner and Skillbred 2012). A suitable material to be used as an interconnect must have high electronic conductivity, mechanical stability, able to operate in high temperature environmental conditions (600-800^{o}C). , has a thermal expansion coefficiment (TEC) or a coefficient of thermal expansion similar to the material used for the electrodes, is easy to fabricate and inexpensive to

manufacture (Werner and Skillbred 2012), (Bash 2015).

Materials that have been used to develop interconnect materials in SOFC applications include Crofer 22 APU, Hitachi ZM6232, Hitachi ZM6232L, Allegheny Ludlum ATI E-Brite, Planse Ducrolloy Cr-5Fe-Y2O3 and sandvik sanergy. (Werner and Skilbred 2012), (Bash 2015),(Amendola et al. 2012),(Alvarez et al. 2011). Apart from sandvik sanergy (SS), these materials do not meet the requirements for a fuel cell system when operating at 700-850°C (Amendola et al. 2012).

The interconnect operating environment is at a high temperature, making the development of interconnect materials continue to be carried out. The development of interconnect materials aimed at creating interconnect materials that can operate at high temperatures, increase mechanical strength and creep resistance, has been carried out by adding Nb to crofer 22. The same approach was carried out on SS. The addition of Nb causes precipitation in the alloy which is known as the laves phase (Werner and Skilbred 2012). SS as a mixture of chromium ferrite metal shows better results such as low oxidation rate, low corrosion rate in various environments. SS also shows a low growth rate and shows increased resistance to oxidation at high temperatures. (Alvarez et al. 2011) High temperature conditions can affect the crystal size of a material (Supriyanto and Holikin 2007). Crystal size initiates powder growth. The increase or decrease in crystal size can be related to the mechanical properties, namely the strength of the alloy produced for interconnect applications. The dominant material with large crystals will reduce durability when given a load (Boskey 2003). Materials with finer grain sizes are able to produce a harder and stronger alloy compared to large coarse grained materials (Mittemeijer and Welzel 2008). Fine grains can be obtained by milling.

Powder milling treatment for 60 hours with a mass ratio of material and ball mill 1:10 showed differences in microstructure that could be observed through SEM morphology (Auger et al. 2013). The advantage of milling treatment is the reduction in particle size (Suryanarayana 2001). The finer the particles because the milling time is too long, will make the distance between the particles smaller and the contact between the particles will increase, allowing agglomeration to occur. Previous studies reported that the milling time of 35 hours showed a decrease in the size of the agglomeration, while the milling treatment of more than 40 hours could destroy the agglomeration smaller powder.

2. Experiments Procedure

Materials

Fe20Cr5AlTi powder is an SS material obtained from Technonoly LTd with a grain size of 106 m which is processed by atomization. The composition of Fe20Cr5AlTi is shown in table 1.

Mechanical alloying condition

The powder milling process is carried out using panetary ball milling produced by MTI Corp. This tool has 4 vials made of alumina with a capacity of 320 mL. The sandvik mass used in this study an amount of 25 g with a mass ratio of SS material with a ball mill of 1:10. Variations in milling time are 0, 8, 24 and 48 hours with a rotation frequency of 30 Hz. The ball mill is driven by rotation and high-frequency vibration.

Table 1. Chemical composition of SS material

Element	Actual (%)
Cr	17.2
Al	6.6
Ti	0.58
Ni	0.47
Mn	0.36
Y	0.097
Cu	0.028
C	0.01
Co	0.01
P	0.005
Fe	Balance (74.64%)

Characterization

SEM (*Scanning Electron Microscope*)

Surface morphology characterization of sandvik powder was carried out using a Hitachi

SU3500 scanning electron microscope (SEM) operated at an acceleration voltage of 20 kV. To get a good morphological picture, 500x magnification was carried out using sofware image J.

XRD (X-Ray Diffraction)

Phases and structures were observed by X-Ray diffraction (XRD) analysis. The sample was scanned using a SmartLab Rigaku XRD with Cu Kα radiation in the range 2θ = 0-80o

PSA (Particle Size Analyzer)

The particle size of the powder was identified with a particle size analyzer (PSA) Cilas 110 dry to get the diameter of the powder.

Crystal Size Calculation

To determine the crystal size of a material, a material diffraction pattern is needed using the Schrerrer equation at the position of the x-ray diffraction peak. x-ray diffraction. The relationship between crystal size and the width of the X-ray diffraction peak can be approximated by the Schrerer equation:

$$D = \frac{\lambda}{B \cos \theta} \qquad (1)$$

where, D is Crystal size (nm), B is Full Width Half Maximum (FWHM). The value used is the FWHM value after being deducted by the instrumental line broadening (radians), θ is Bragg's angle notation, λ is X-Ray wave length notation. To obtain a more accurate estimate of the crystal size results, the FWHM value must be corrected by instrumental line broadening based on the equation:

$$B = \sqrt{FWHM\ Sample - FWHM\ standard}$$

where FWHM is the width of the diffraction peak at half the maximum of the test sample and the standard FWHM is the peak width of a very large standard material, the peak is around the location of the peak of the sample to be calculated.

3. Result and Discussion

Characterization

The results of the XRD data in Figure 1 are the relationship between the intensity on the x-axis and the diffraction angle (2θ) on the y-axis. The diffraction peak that appears shows a crystal plane which indicates the presence of the Cr0.5 Fe0.5 phase. The Fe-Cr phase was dominant at each milling time indicated by the high peak intensity.

The decrease in intensity indicates the calculation of the crystal size obtained is getting smaller. This happens because milling aims to mix the material homogeneously or reduce the grain size of the powder; The ball mill is driven by rotation and high-frequency vibration. The rotational movement or vibration of the ball mill produces a centrifugal force when the vial rotates on its axis. (Suryanarayana 2001) As a result, the trapped material between the ball mill and the vial wall will collide with each other resulting in a change in the shape of the material so that it is split into smaller arrangements. (B.Huang 1995).

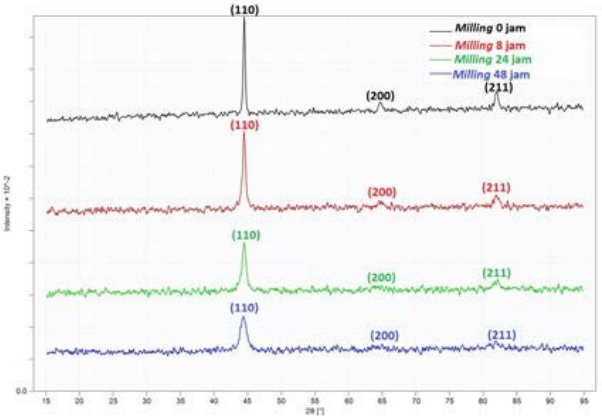

Figure 1. Sanergy sandvik diffractogram with milling time

Crystal size can be calculated by calculating FWHM from XRD results with Scherer's formula. Based on this method, the smaller the crystal size, the wider the x-ray diffraction peak produced. (Monshi 2012) Figure 2 shows the longer the SS milling treatment time, the greater the FWHM value, the wider the x-ray diffraction peak that appears and the resulting intensity decreases. This affects the smaller crystal size as the FWHM value increases.

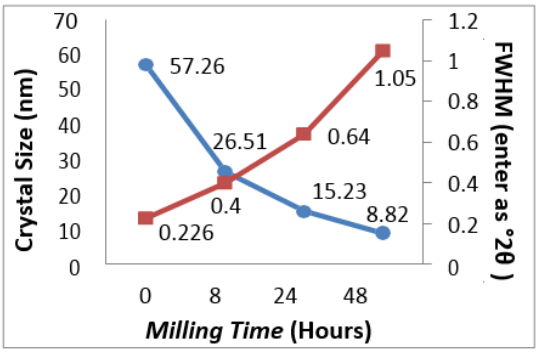

Figure 2. Graph of crystal size calculation

SEM Analysis

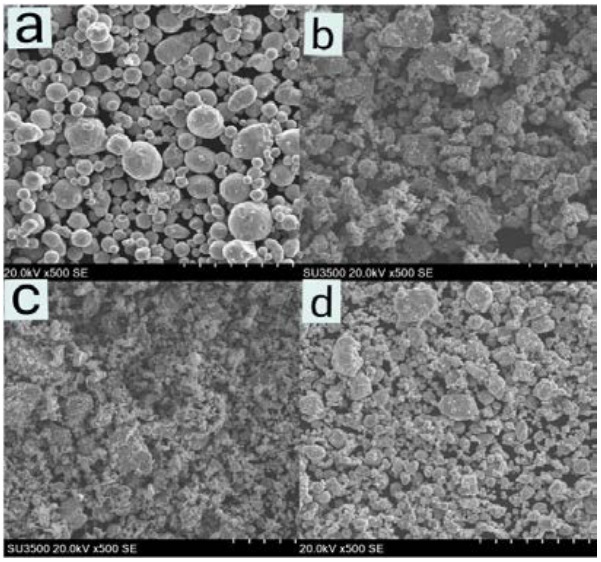

Figure 3. Morphology of SS powder with milling time (a) 0, (b) 8, (c) 24, and (d) 48 hours

Grain size can also be observed based on SEM morphology (Figure 3). Grain size in the figure (a) there is a clear grain boundary with a large size of 15 m, while after 8 hours of milling treatment (Fig. b) it appears that the grain no longer has a clear boundary, the grain size is 7 m, (Fig.c) it appears that the grains are no longer intact and it is clear that the grain size is experiencing agglomeration, the grain size is which is measured at 10μm, after adding the milling treatment time, the grain size appears smaller to 5μm. Milling with very fine particles will cause the coupling forces to become larger and the presence of chemical bonds or Van Der Waals forces with bond strengths of 40-400 kJ/mol can make the particles coalesce or agglomerate. If there are particles trapped and then given an impact force, the particles can agglomerate (Ullah et al. 2014).

Table 2. PSA measurement results

Milling Time (Hours)	Diameter Partikel (μm)
0	36.79
8	11.83
24	20.13
48	17.68

The results of PSA Table 2. it can be seen that the particle diameter at 24 hours milling is larger than the previous milling time, this is due to agglomeration as shown in the SEM morphology Figure 3.

4. Conclusion

From the characterization results, it can be concluded that increasing milling time shows the phenomenon of lower X-ray intensity, widening peak intensity, and physical properties, namely decreasing crystal size. The grain morphology on SEM and PSA showed the same phenomenon, at 24 hours milling showed agglomeration causing the particle size to increase. The smallest crystal size obtained at milling time of 48 hours, can be applied as a study of SOFC interconnect materials.

Acknowledgment

Thank you to the Physics Research Center of the Indonesian Institute of Sciences for the research funds provided through the thematic DIPA in 2020.

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