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# Thermal expansion, porosity, and microhardness properties of solid oxide fuel cell metallic interconnects manufactured by powder metallurgy approach

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## ABSTRACT

The effects of manufacturing parameters on the physical, thermal and mechanical properties of solid oxide fuel cell (SOFC) metallic interconnects manufactured through the powder metallurgy (P/M) method were investigated. To this goal, interconnect samples were first fabricated through the P/M technique using Nickel, Stainless steel 316L, Inconel 600, SUS 445J1, 1C44Mo20, and Crofer®22 APU powders. Varied manufacturing parameters (compaction pressure, compaction temperature, and sintering temperature) were adopted to obtain sound samples. For characterization purposes, porosity, microhardness, and coefficient of thermal expansion (CTE) measurements were performed on the samples. Results showed that the porosity and CTE values of samples decreased with the increasing compaction pressure and temperature as well as sintering temperature while microhardness values increased. It was concluded that only the Crofer®22 APU powders satisfied the coefficient of thermal expansion requirement for SOFCs suggested in the literature.

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## 1. INTRODUCTION

Solid oxide fuel cells (SOFCs) are highly efficient energy conversion devices used as a power supply or electricity generator for stationary applications [1]. The main advantages of the SOFC system are operating at high temperatures (600-1000 °C), not requiring an expensive catalyst layer, fuel flexibility, and being appropriate for cogeneration applications [2, 3]. A SOFC stack consists of membrane electrolyte assembly (MEA), sealant, and interconnect components. Today, yttria-stabilized zirconia (YSZ) based ceramic materials are used as MEA, and glass-ceramic materials using as a sealant in SOFC systems generally [4, 5]. Interconnect is one of the critical components of SOFCs stack through which multiple cells are connected in series [6]. Moreover, interconnects provide electrical contacts between cells, distribute reactive gases on both sides of the cell (anode and cathode sides), and separate the anodes and cathodes of adjacent cells in the stack [6-8]. Interconnects are usually made from stainless steel materials due to their excellent features such as high electrically and thermally conductivity, corrosion resistance, and high-density structure. Interconnects are manufactured using casting and machining (wire erosion) operations in general [9, 10]. On the other hand, they can be manufactured using the powder metallurgy (P/M) method. The P/M approach has some advantages over traditional manufacturing, such as reducing machining steps and scrap material and near-net-shape production [11, 12]. The porous Ti – 5.4% Si material was produced by powder metallurgy method studied by Brodnikovskii et al. [13] and examined its structural and mechanical properties. Different groups carried out researches about metallic interconnect manufacturing by powder metallurgy approach [14-16]. Glatz et al. [17-19], Köck et al. [20], and Janousek et al. [21] manufactured different net-shaped interconnect materials with the P/M method. In addition, they indicated that the powder metallurgy method is more comfortable than the traditional manufacturing process, and manufactured interconnects by P/M were appropriate for interconnect application in the SOFC system. Öztürk et al. researched the oxidation, electrical, and mechanical properties [22] and fuel cell performance [23] of the Crofer®22 APU interconnects manufactured via the P/M method and compared their features with the commercial bulk form of the same material.

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Although the P/M Crofer®22 APU interconnect was contacted a better interface with glass-ceramic sealant, its performance was lower than the bulk interconnects due to exposure oxidation at the grain boundaries. In another study by the same group, the effects of P/M manufacturing processes on the ferritic Fe22Cr steel material were investigated [24]. Porosity and thermal expansion coefficient decreased with the increase of production parameters. Also, it was noted that the oxidation behaviour of the P/M material was influenced not only by process parameters but also by powder shape.

In this study, some metallic interconnect powders used as interconnect in literature were manufactured by the P/M method and investigated whether appropriate as interconnect application for the SOFC systems. In different manufacturing parameters, samples were fabricated from nickel, stainless steel 316L, Inconel 600, SUS 445J1, 1C44Mo20, and Crofer®22 APU powders. Afterwards, the effects of manufacturing parameters on CTE, porosity, and microhardness were scrutinized.

## 2. EXPERIMENTAL

Interconnect powders were acquired from different countries and companies. Physical specifications and chemical compositions of powders are listed in Table 1 and Table 2, respectively. Also, SEM images of powders are presented in Figure 1.

Powder size significantly affects interconnect manufacturing by the P/M method as it directly affects the adhesion surfaces and porosity during compaction. The melting temperatures of the powders are one of the most critical parameters to be considered during sintering. Powders consist of iron-based materials, as seen in Table 2. At the same time, the chromium ratio is very high without ‘Nickel’ powder. Chromium additives increase chemical stability, oxidation resistance, and anti-corrosion levels. Sample manufacturing parameters are given in Table 3. Compaction temperature was considered 300, 375, and 450 °C (warm pressing conditions). Compaction pressure was varied at the range of 200-400 MPa. Moreover, 900, 1050, and 1200 °C sintering temperatures were evaluated. Hydraulic press (60 tons capacity) and die set (have 30 mm$^2$ areas) mechanism as shown in Figure 2 was used in sample manufacturing with the P/M approach.

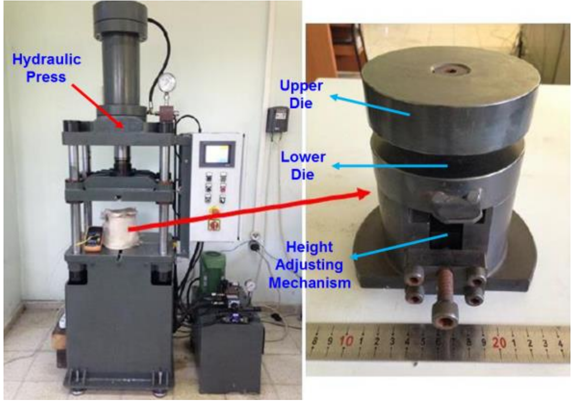


Fig. 2. Hydraulic press and die set used in sample manufacturing.

Table 1. Physical specifications of powders.

| *Metal Powder* | *Particle Size Distribution (µm)* | *Density (g/cm$^3$)* | *Melting Temperature (°C)* |
|---|---|---|---|
| Nickel | 0-125 | 8.9 | 1455 |
| Stainless Steel 316L | 0-125 | 8 | 1400 |
| Inconel 600 | 0-125 | 8.4 | 1350 |
| SUS 445J1 | 0-58 | 7.7 | 1500 |
| 1C44Mo20 | 0-38 | 7.9 | 1490 |
| Crofer®22 APU | 0-63 | 7.7 | 1510 |

### 2.1. Porosity Measurements

Porosity values affect the coefficient of thermal expansion (CTE) directly. At the same time, interconnects should dense as possible because reactant and oxidant gases can pass from interconnect to outside. So, firstly porosity values of powders were determined. Samples were moulded using the cold moulding method, and grinding and polishing processes were carried out. Microscope images of samples were obtained, and then porosity values were determined using ImageJ software. Porosity measurement process steps are given in Figure 3. Firstly, the original microscope image was uploaded to the software, as seen in Figure 3 (a). This image was converted into black and white areas (binarization), as seen in Figure 3 (b). Then, black and white areas as seen in Figure 3 (c) were selected as in red rectangle areas. The ratio of black on all areas was determined, and the porosity value was calculated.

### 2.2. Microhardness Measurements

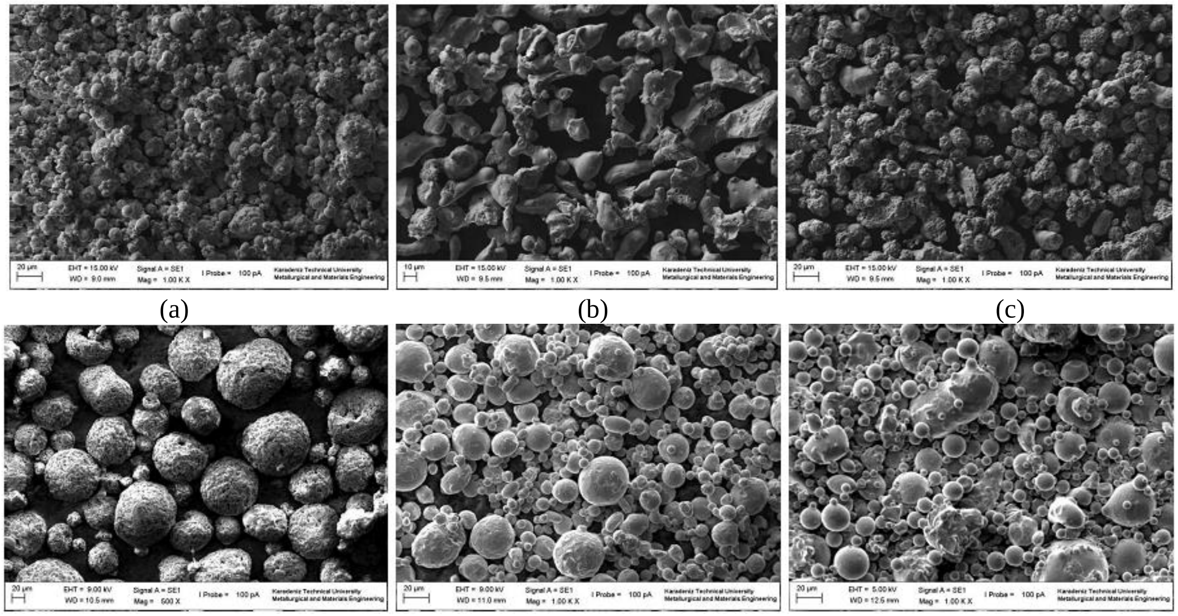
Interconnects should be strengthened mechanically because they expose mechanical loads under operating conditions. Vickers microhardness measurements were carried out using the Innova microhardness test device. 50 g.f load was applied at a dwell time of 10 s. Measurements were saved from five different points on a sample. Randomly selected microhardness measurement points (1, 2, 3, 4, 5 points on the figure) of a sample are shown in Figure 4.

### 2.3. CTE Measurements

Coefficient of thermal expansion (CTE) values of SOFC system components should close and match as possible. Usually, the CTE of system components changes between 9-12 x 10$^{-6}$ K$^{-1}$ [25]. Thus, CTE values of interconnecting should match these values. Besides, differences in the CTE of components can cause thermal stresses and cracks in the system [26]. For this purpose, samples were prepared with 20x10x3 mm$^3$ dimensions by the P/M method. Measurements were carried out using a dilatometer device. Samples were heated from room temperature to 800 °C, and CTE was measured at this point.

Table 2. Chemical compositions of powders.

| *Element % (wt)* | *Ni* | *Fe* | *Cr* | *Mo* | *Mn* | *Si* | *Ti* | *Nb* | *Mn* | *La* | *Other* |
|---|---|---|---|---|---|---|---|---|---|---|---|
| *Nickel* | 99,8 | - | - | - | - | - | - | - | - | - | 0,2 |
| *Stainless Steel 316L* | 10-14 | 67,5 | 17 | 2,5 | 0-2 | - | - | - | - | - | - |
| *Inconel 600* | 72 | 6-10 | 14-17 | - | - | - | - | - | - | - | - |
| *SUS 445J1* | 0,09 | Bal. | 22,3 | 1,2 | 0,08 | 0,28 | 0,19 | 0,26 | 0,1 | - | - |
| *1C44Mo20* | 0,02 | Bal. | 22,1 | 1,0 | 0,31 | 0,04 | 0,02 | 0,73 | - | 0,1 | - |
| *Crofer®22 APU* | 0,03 | Bal. | 22,8 | 0,1 | 0,44 | 0,5 | 0,2 | 0,1 | - | 0,1 | - |


(a) (b) (c)

(d) (e) (f)

Fig. 1. SEM images of powders; (a) Nickel, (b) Stainless steel 316L, (c) Inconel 600, (d) Stainless steel SUS 445J1, (e) 1C44Mo20, (f) Crofer®22 APU.

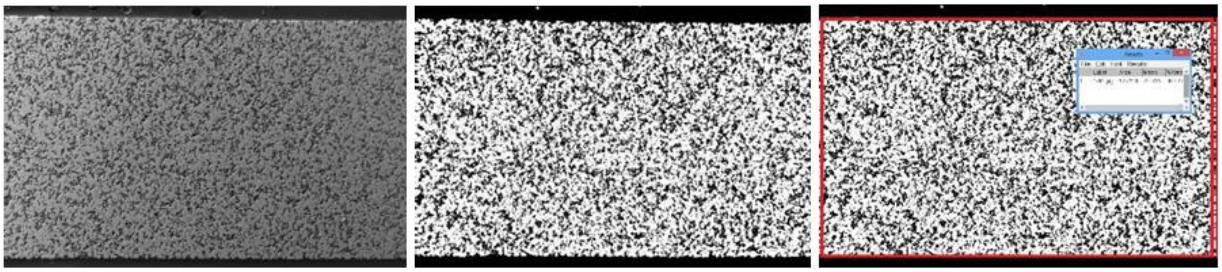

(a) (b) (c)

Fig. 3. Porosity measurements using Image J software; (a) original microscope image, (b) binarization, (c) determining the ratio of black or white areas to the whole specified area (red boundaries)

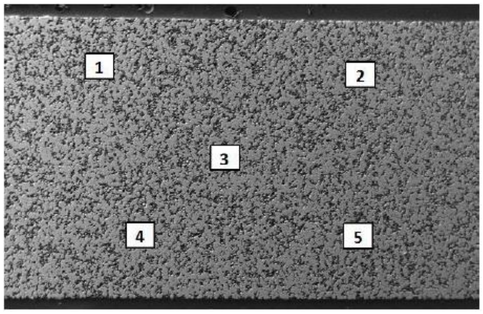


Fig. 4. Microhardness measurement points.

## 3. RESULTS

### *3.1. Porosity Values*

The porosity value was found for Sample #101 as 7.4% and Sample #109 as 3.2% for Nickel powders. Porosity values decrease with the increase of production pressure and temperature for all powders. The highest porosity value for the stainless steel 316L sample was 11%, with Sample #201. The lowest porosity for the same group was calculated as 5.8% with Sample #209. The same trends were observed for other powder groups. The porosity values of all samples are presented in Figure 5. The mentioned temperature and pressure legends of the x-axis in Figure 5 and Figure 6 belong to the manufacturing parameters. Sintering temperature was implied with coloured square indicators in the same figures. 900, 1050, and 1200 °C sintering temperatures were depicted with brown, orange, and pink colours, respectively. The Crofer®22 APU powder was observed as the most porous sample, while Nickel powder has the lowest porosity value. Therefore, porosity is a natural result of the powder metallurgy approach and can control the changing of manufacturing parameters (sintering temperature, compaction pressure, and compaction temperature) [27]. Sotomayor et al. analyzed the mechanical characterization of the 430L stainless steel porous supports obtained via powder extrusion moulding approach [27]. They stated that the sintered samples with 35% porosity are suitable for SOFC to interconnect application. Besides, the porosity affects the properties of the material. Antepara et al. [28] fabricated porous substrates by the P/M method using Crofer powders and investigated the oxidation resistance. They noted that the lower porosity (30 vs 70%) positively affects the oxidation resistance because of the lower surface area. In another study by Antepara et al. [29], it was reported that the electrical resistivity (ASR) measurements were not conducted to P/M interconnects due to the high oxidation level of the samples.

### *3.2. Microhardness Values*

Microhardness values of samples are presented in Table 4. Microhardness values increased with increased production pressure, temperature and sintering temperature. It can be concluded that the compacted samples became more resistant to penetration during the test with the increasing fabrication parameters [30]. In other words, the effective mechanism is powder deformation in low porosity interconnects. Thus, it causes an increase in microhardness [31].

All interconnect candidates were found adequate in terms of mechanical strengthen. Even though the compacted powders have insufficient endurance and fragile structure, the interconnect candidates have gained mechanical durability after the sintering process [32]. Acchar et al. manufactured ceramic interconnected using $La_{0.80}Sr_{0.20}Cr_{0.92}Co_{0.08}O_3$ powders and reported that the dense (lower porosity) samples had higher hardness and strength values [32].

Table 3. Sample manufacturing parameters.

| *Metal Powder* | *Sample Code #* | *Compaction Temperature (°C)* | *Compaction Pressure (MPa)* | *Sintering Temperature (°C)* |
|---|---|---|---|---|
| Nickel | 101 | 300 | 200 | 1200 |
| | 102 | 300 | 300 | 1200 |
| | 103 | 300 | 400 | 1200 |
| | 104 | 375 | 200 | 1200 |
| | 105 | 375 | 300 | 1200 |
| | 106 | 375 | 400 | 1200 |
| | 107 | 450 | 200 | 1200 |
| | 108 | 450 | 300 | 1200 |
| | 109 | 450 | 400 | 1200 |
| Stainless Steel 316L | 201 | 300 | 200 | 1200 |
| | 202 | 300 | 300 | 1200 |
| | 203 | 300 | 400 | 1200 |
| | 204 | 375 | 200 | 1200 |
| | 205 | 375 | 300 | 1200 |
| | 206 | 375 | 400 | 1200 |
| | 207 | 450 | 200 | 1200 |
| | 208 | 450 | 300 | 1200 |
| | 209 | 450 | 400 | 1200 |
| Inconel 600 | 301 | 300 | 200 | 1200 |
| | 302 | 300 | 300 | 1200 |
| | 303 | 300 | 400 | 1200 |
| | 304 | 375 | 200 | 1200 |
| | 305 | 375 | 300 | 1200 |
| | 306 | 375 | 400 | 1200 |
| | 307 | 450 | 200 | 1200 |
| | 308 | 450 | 300 | 1200 |
| | 309 | 450 | 400 | 1200 |
| SUS 445J1 | 401 | 300 | 200 | 900 |
| | 402 | 375 | 200 | 1050 |
| | 403 | 450 | 200 | 1200 |
| | 404 | 300 | 300 | 900 |
| | 405 | 375 | 300 | 1050 |
| | 406 | 450 | 300 | 1200 |
| | 407 | 300 | 400 | 900 |
| | 408 | 375 | 400 | 1050 |
| | 409 | 450 | 400 | 1200 |
| 1C44Mo20 | 501 | 300 | 200 | 900 |
| | 502 | 375 | 200 | 1050 |
| | 503 | 450 | 200 | 1200 |
| | 504 | 300 | 300 | 900 |
| | 505 | 375 | 300 | 1050 |
| | 506 | 450 | 300 | 1200 |
| | 507 | 300 | 400 | 900 |
| | 508 | 375 | 400 | 1050 |
| | 509 | 450 | 400 | 1200 |
| Crofer®22 APU | 601 | 300 | 200 | 900 |
| | 602 | 300 | 300 | 1050 |
| | 603 | 300 | 400 | 1200 |
| | 604 | 375 | 200 | 900 |
| | 605 | 375 | 300 | 1050 |
| | 606 | 375 | 400 | 1200 |
| | 607 | 450 | 200 | 900 |
| | 608 | 450 | 300 | 1050 |
| | 609 | 450 | 400 | 1200 |

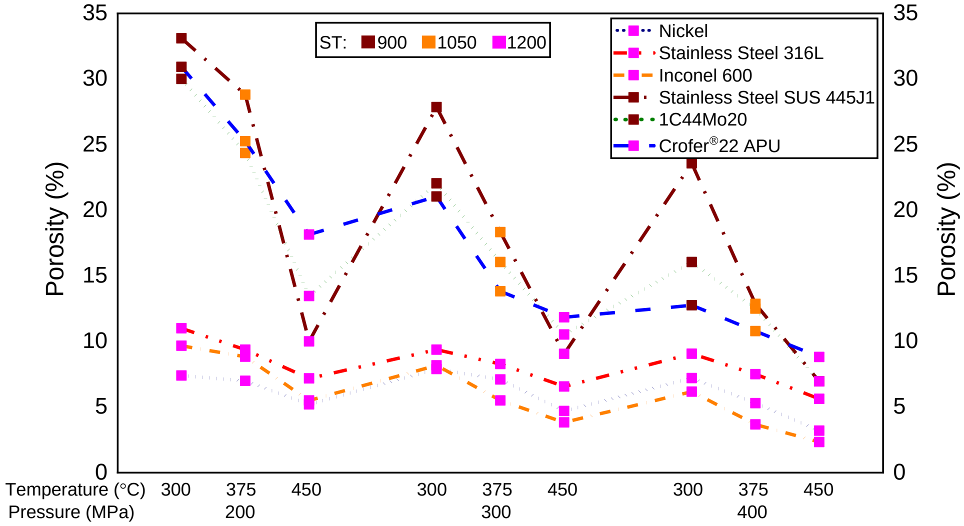


Fig. 5. Variation of porosity values of the samples with the increase of compaction temperature and pressure

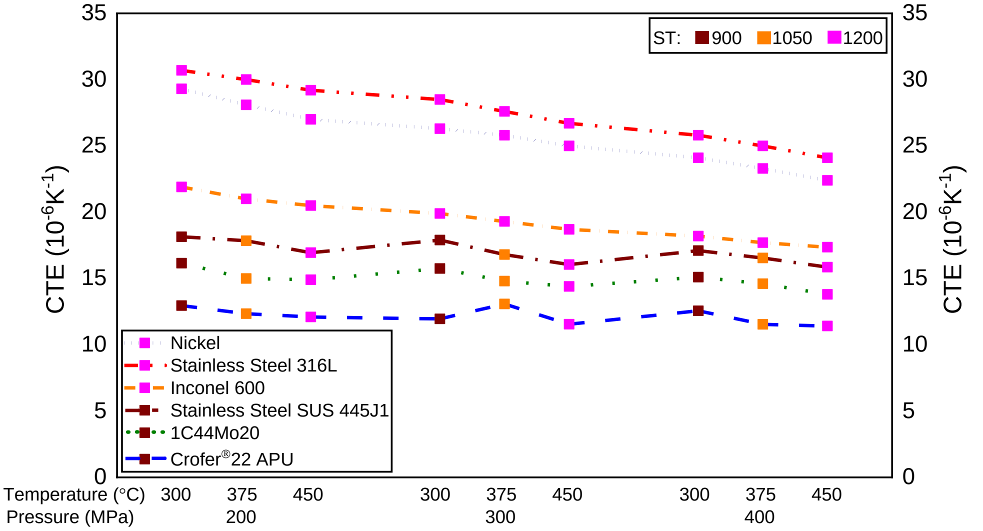


Fig. 6. Variation of the CTE values with respect to change in compaction temperature and pressure

The bonding strength between a glass-ceramic and Crofer 22 APU interconnect was investigated by Sharma and Singh [33], and a microhardness value of 384 HV was reported. SOFC stacks operate under compressive loads, and each component of the system should satisfy required mechanical expectations. In this regard, microhardness values of the P/M interconnect were found lower than that for bulk form as resistance against indentation can be lower for porous surfaces. Nevertheless, the microhardness levels of all P/M samples were found sufficient for the interconnect application.

### 3.3. CTE Values

CTE values of the samples were found to decrease with the increase in compaction pressure and temperature. CTE of Nickel samples was found in the range of 22-29x10$^{-6}$ K$^{-1}$. On the other hand, CTE of 316L stainless steel 316L samples was measured in between 24 - 31x10$^{-6}$ K$^{-1}$. CTE values of Inconel 600 samples were noted in the range of 18 - 22x10$^{-6}$ K$^{-1}$. The smallest CTE value for SUS 445J1 stainless steel was recorded as 15.82x10$^{-6}$ K$^{-1}$ (sample #409). The lowest CTE value for 1C44Mo20 samples was 13.82x10$^{-6}$ K$^{-1}$ (sample #509).

CTE values of all samples were found to mismatch for the SOFC system, except the Crofer®22 APU powders. CTE value variation was obtained for Crofer®22 APU powders in the 11.42-13.08x10$^{-6}$ K$^{-1}$ range. Thus, only Crofer®22 APU is appropriate for interconnect application for the SOFC system. Variation of CTE values for all the interconnect samples fabricated with respect to manufacturing temperature and pressure were illustrated in Figure 6.

Figure 5 and Figure 6 have comparable specifications. As it can be noticed from these figures, the Crofer®22 APU sample has the highest porosity value and the lowest CTE concurrently. On the contrary, the 316L stainless steel sample has lower porosity than samples produced with other powders, while the sample has the highest CTE. In addition, the changes in porosity levels are higher compared to those. This is attributed to the fact that CTE is an intrinsic material property while porosity can be varied significantly with the production parameters.

Table 3. Microhardness values of samples.

| *Metal Powder* | *Sample Code #* | *Microhardness (HV$_{0.05}$)* |
|---|---|---|
| Nickel | 101 | 104.4 |
| | 102 | 113.1 |
| | 103 | 121.4 |
| | 104 | 107.0 |
| | 105 | 115.6 |
| | 106 | 123.5 |
| | 107 | 108.4 |
| | 108 | 118.2 |
| | 109 | 125.9 |
| Stainless Steel 316L | 201 | 134.3 |
| | 202 | 139.5 |
| | 203 | 142.8 |
| | 204 | 145.4 |
| | 205 | 151.1 |
| | 206 | 159.6 |
| | 207 | 162.3 |
| | 208 | 175.4 |
| | 209 | 178.6 |
| Inconel 600 | 301 | 123.7 |
| | 302 | 125.2 |
| | 303 | 128.4 |
| | 304 | 127.3 |
| | 305 | 130.6 |
| | 306 | 133.8 |
| | 307 | 131.7 |
| | 308 | 134.9 |
| | 309 | 136.3 |
| SUS 445J1 | 401 | 111.4 |
| | 402 | 116.8 |
| | 403 | 121.1 |
| | 404 | 126.5 |
| | 405 | 134.9 |
| | 406 | 144.3 |
| | 407 | 146.8 |
| | 408 | 149.4 |
| | 409 | 151.2 |
| 1C44Mo20 | 501 | 129.1 |
| | 502 | 147.4 |
| | 503 | 153.6 |
| | 504 | 136.3 |
| | 505 | 167.6 |
| | 506 | 173.8 |
| | 507 | 219.7 |
| | 508 | 234.3 |
| | 509 | 241.2 |

## 4. CONCLUSION

In this study, some metallic interconnects were manufactured by the P/M approach, and their suitability for SOFC working conditions was investigated. Samples were first manufactured with different compaction pressure, compaction temperature, and sintering temperature. Effects of these production parameters on porosity, microhardness, and CTE values of powders were investigated. Results showed that the porosity and CTE values of samples decreased with the increasing compaction pressure and temperature as well as sintering temperature while microhardness values increased. All samples have sufficient mechanical strength based on microhardness test results. Besides, the coefficient of thermal expansion of Crofer$^{®}$22 APU powders was found to be compatible with the CTE of other components of SOFC (9-12x10$^{-6}$ K$^{-1}$) while the other powders (14-31x10$^{-6}$ K$^{-1}$) were not.

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