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ORIGINAL ARTICLE

Phase analysis and microstructure study of sintered Ni-Cr composites containing MoS_2, Ag and CaF_2 additives as solid lubricants

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ABSTRACT – Ni-Cr based composites with and without the addition of solid lubricants (MoS_2, Ag and CaF_2) were prepared by powder metallurgy method. The samples were sintered at different temperatures (1000°C, 1100°C and 1200°C) under controlled atmosphere and various holding times. The physical properties such as sintered density, relative density and porosity were studied. The microstructures and phase studies of the Ni-Cr based composites were conducted using Scanning Electron Microscope (SEM) and X-Ray Diffraction Analysis (XRD) while the hardness of the composites was measured by the Vickers Micro Hardness Tester. The results revealed that as the sintering temperature was increased, the sintered density, relative density and hardness also increased while the porosity was reduced. At the sintering temperature of 1200°C, the relative density reached its maximum value. SEM and XRD analysis confirmed the existence of MoS_2 and Ag in sintered samples. MoS_2 and Ag were not mainly dissolved and/or decomposed even after sintering at 1200°C.

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KEYWORDS
Self-lubricating composites; powder metallurgy; solid lubricants; Ni-Cr composites; MoS_2; Ag; CaF_2.

INTRODUCTION

Rapid development in advanced technologies requires most of the mechanical components to work at high temperature. As for example, the turbine engines used in the aviation industries and the mechanical devices in operating over a wide range of temperature [1, 2]. The machine elements such as bearings and bushes are subjected to friction and wear at high temperature which leads to mechanical failure. There is a high maintenance cost attached to these heavy-duty applications due to wear and oxidation [3]. Instead of using traditional liquid lubricants, the solid lubricants are preferable due to their advantages such as better lubrication and higher chemical stability at elevated temperatures; good dimensional stability that allows precision finishing to high accuracy; ability to function without cooling, thus eliminating cooling systems; and no seals are required to isolate them from the process fluid [4].

Self-lubricating composites have the ability of self-lubrication during movement between two surfaces due to the presence of soft malleable phase at the interface between the sliding surface in order to reduce friction and wear [5]. Self-lubricating composites are produced through powder metallurgy process since several decades. The particles of solid lubricant can be easily inserted in the matrix during the mixing process [6]. Therefore, process is able to modify the materials microstructure and obtained desired properties and performance [7, 8]. The usage of Ni-Cr self-lubricating composites has been widely accepted in applications such as gas turbine engines, aircraft, marine and also petrochemical plants [9].

Research is continuously done to improve the Ni-Cr based composites in term of their wear durability and mechanical strength. The addition of solid lubricants such as MoS_2, Ag and CaF_2 is expected to reduce the friction and wear of the composites [8]. The usage of more than single solid lubricant is preferable because no single solid lubricant can act on a wide range of temperatures [9, 10]. As for example, MoS_2 is known as the low-temperature solid lubricant. It can decompose and oxidize during use at temperatures greater than 300°C [11, 12]. Soft metal such as Ag is used as mid-temperature range solid lubricant, which is between 300 to 500°C where it can form a thin film on the worn surface due to the plastic formation. Above 500°C, excessive softening tend to occur that affects the efficiency of the surface lubrication through solid lubricants [13, 14]. For high-temperature lubrication systems above 400°C, fluorides have been tested (BaF_2/CaF_2). These fluorides undergo brittle to ductile transformation, forming a fully ductile phase with very low shear strength [15, 16]. However, the addition of solid lubricants has to be optimized as the addition of solid lubricants may increase the surface properties as well as reduce the mechanical strength of the composite. This should be noted that since surface lubrication is a very superficial phenomenon, it merely depends on surface temperature during sliding, which may be different from the overall temperature of the component under a sliding motion [17, 18].

Therefore, research has been conducted continuously to improve and produce a combination of matrix material and solid lubricant/s that can perform well in the wide range of temperatures as well as provide good mechanical strength and surface properties. The present research is to study the sintering process of Ni-Cr alloy based composites when several

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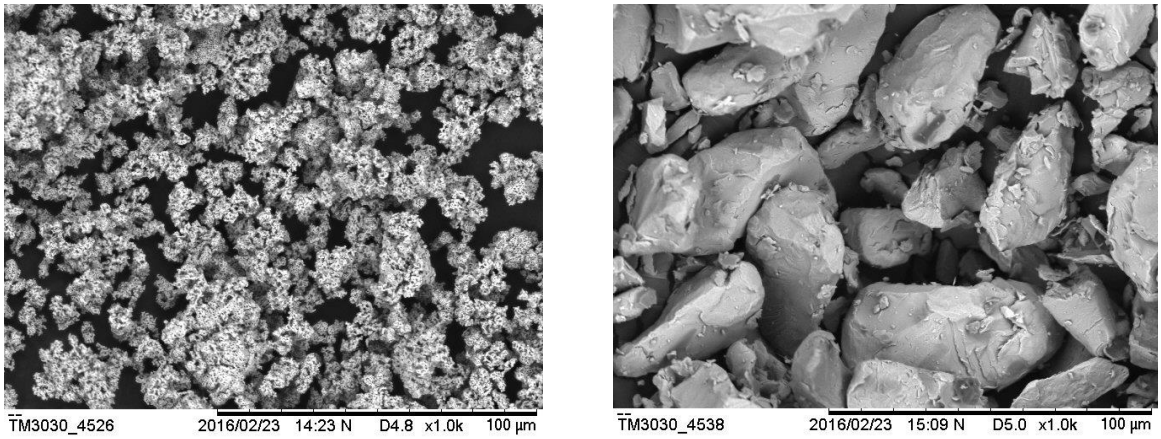
solid lubricants in different combination and quantity are added into the system. The main discussion point is the optimization of sintering cycle when Ni-Cr is combined with important quantity of solid lubricants.

MATERIALS AND METHODS

Sample preparation

The Nickel- Chromium (Ni-Cr) composites contain 80% of Ni and 20% of Cr as base alloy in which molybdenum disulphide (MoS_2), silver (Ag) and calcium fluoride (CaF_2) are added as solid lubricants. The powders are manufactured by Sigma Aldrich Germany. The characterizations of the powders are done using a Scanning Electron Microscope (SEM) by Hitachi TM3030 and X-Ray Fluorescence (XRF) by Bruker S4 Pioneer, USA. SEM analysis is conducted to observe the particle shape and distribution while XRF analysis is conducted to determine the purity of the powder [20]. Figure 1 (a) and (b) shows the SEM micrograph of the nickel and chromium powders.



(a) (b)

Figure 1. (a) Powder morphology of nickel and (b) Powder morphology of chromium.

In Figure 1 (a), SEM image at 1000X illustrates that nickel has a dendritic particle shape. Such shape represents a synthesis process based on chemical precipitation. The purity of the nickel powder is 99.1% determined by XRF analysis. Figure 1 (b) shows the particle shape and distribution of chromium powder. The particle shape and size indicates the mechanical attrition process. Smaller particles attached to the bigger particles may be produced due to the ball milling during the production process. XRF analysis determines the purity of chromium powder as 93.3%, which is acceptable for such mechanically milled powder. Figures 2-4 show the SEM images of the microstructure of solid lubricants which are MoS_2, Ag and CaF_2.

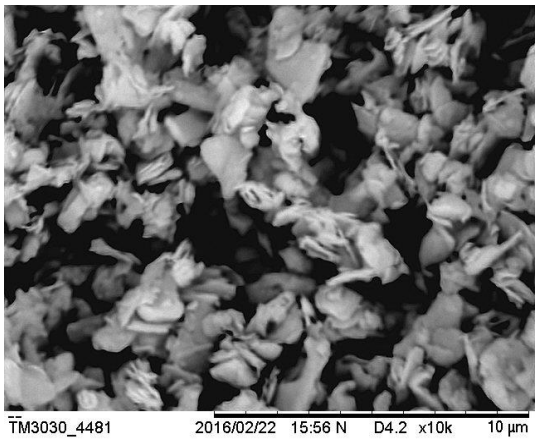


Figure 2. Powder morphology of MoS_2

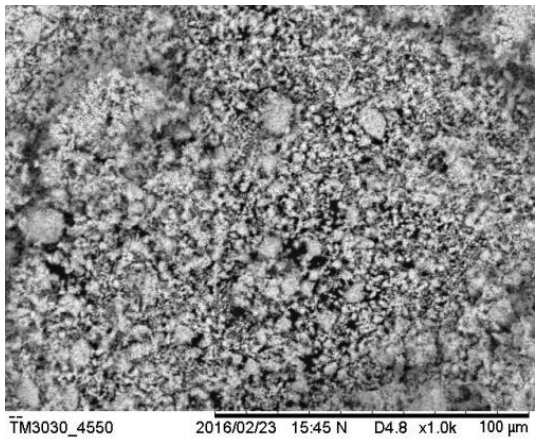


Figure 3. Powder morphology of Ag

Figure 2 demonstrates the particle shape of MoS_2 powder which is in flake shape. The purity of the powder is Mo 72% and S 25.1%. SEM microstructure presented in Figure 3 shows the particle distribution of nanosilver powder.

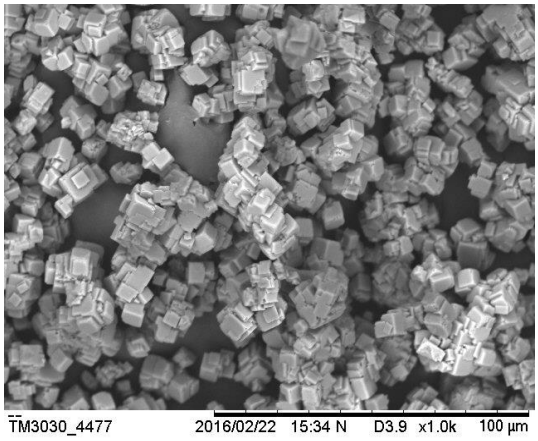


Figure 4. Powder morphology of CaF_2.

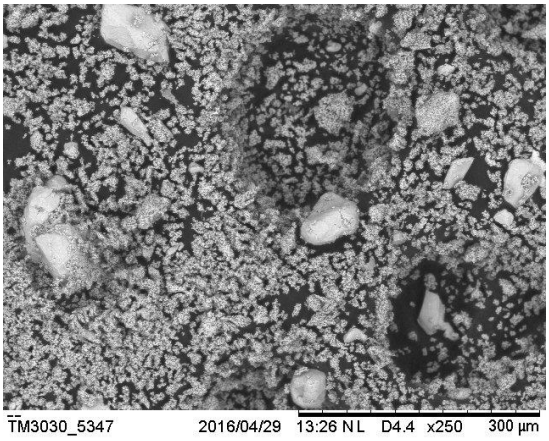


Figure 5. Powder morphology of Ni-Cr.

Figure 4 presents the particle size and shape of CaF_2 powder where it can be seen that the shape is cuboidal crystal like which demonstrates an evaporation-condensation stage during the synthesis of the powder. The micrograph illustrates the stacked crystallized structure of CaF_2. The purity of this powder is Ca 99.1% verified by XRF analysis. XRD analysis is also conducted to confirm the existence of CaF_2.

Ni-Cr based composites were produced through powder metallurgy method which consists of mixing, cold compaction and sintering. Various denominations of the mixtures are given in Table 1. The powder was weighed by analytical balance (Mettler, USA) and mixed in indicated quantities. Table 1 shows the composition of Ni-Cr based composites.

Table 1. Composition of Ni-Cr based composites.

Composite	Designation	Composition (wt%)			
		80Ni-20Cr	MoS_2	Ag	CaF_2
1	NC	Balance	0	0	0
2	NCM	Balance	5	0	0
3	NCA	Balance	0	5	0
4	NCCf	Balance	0	0	5

The powder mixture was mixed homogeneously, in dry condition, in a ball mill (US Stoneware, Model:764 AVM) with the speed of 300rpm for 30minutes. SEM micrograph of mixed powder is shown in Figure 5. The mixture of powder was compacted using a cold compression machine (ENERPAC) with the compaction pressure of 700MPa. The compacted samples were in a pellet shape (CARVER, 13mm dia ID Pellet Die) with the thickness of 5mm. The compacted samples were sintered in a high-temperature furnace (Brand: MTI, Model: GSL – 1700X) for 60 minutes in an argon atmosphere at temperatures of 1000ºC, 1100ºC and 1200ºC. The heating and cooling rate was kept at 10ºC/min.

Figure 5 shows the distribution of Nickel and Chromium powder after mixing by using the ball mill machine for 30 minutes. The bigger particles represent chromium powder while the smaller particles represent nickel powder. The mixture was homogeneously distributed throughout the process. Chromium particles may have broken into smaller size during the milling process while some nickel particles tend to agglomerate on the chromium surface. The mixture was compacted after milling by a hydraulic press into a pellet.

The diameter and height of the green and sintered samples were measured by using vernier caliper while the mass of the samples was obtained by the analytical balance. The readings were taken three times and average values were taken. The density and porosity of the samples were calculated using the geometric method using mixture formula. The formula to calculate the density $\rho = m / V$ (m = mass of pellet, V = volume of pellet) meanwhile the porosity was measured by the formula of $(\rho_{theoretical} - \rho_{actual}) \times 100 / \rho_{theoretical}$. The hardness of Ni-Cr based composites was measured by using Shimadzu Micro Hardness Machine with the diamond indenter at an indentation load of 9.807 N with the dwell time of 10s. For surface preparation, samples were ground and polished using Grinder and Polisher machine. The microstructure of the sintered samples was observed and analyzed by using Table-Top Scanning Electron Microscope (SEM-TM3030). The phase analysis was done by using Field Emission Scanning Electron Microscope (AMARY 1000 FESEM) attached to Energy Dispersive X-Ray (EDX). The elemental and weight percentage analysis conducted by EDX. Phase analysis was also performed using X-ray Diffraction analysis (XRD) on a Bruker AXS D8 Advanced Diffractometer.

RESULTS AND DISCUSSION

Sintered Density, Porosity and Hardness.

The effect of sintering temperature on the sintered density, porosity and hardness of Ni-Cr based composites was investigated. The composites were sintered at 1000, 1100 and 1200ºC for 1 hour in the argon gas atmosphere. The effect of sintering temperature on the sintered density, porosity and hardness of the composites is shown in Table 2.

Table 2. Sintered density, porosity and hardness of Ni-Cr based composites at different sintering temperature.

Temp. / Samples	Sintered Density (g/cm^3)			Porosity (%)			Hardness Load= 9.807N (HV)		
	1000ºC	1100ºC	1200ºC	1000ºC	1100ºC	1200ºC	1000ºC	1100ºC	1200ºC
NC	5.59	6.08	6.99	35	29	18	57.6	62.18	147.9
NCM	5.67	6.25	6.93	32	25	17	50.28	54.34	112.1
NCA	6.07	6.35	7.36	30	27	15	53.39	60.66	113.0
NCCf	5.91	6.30	7.17	29	24	13	62.17	69.52	114.6

Based on Table 2, the sintered density of all composites is increasing as the sintering temperature is increasing. The highest sintered density was achieved when the sintering temperature was at 1200ºC for all composites. At higher sintering temperature, the diffusion rate is higher which makes the material transport through vacancies easier and thus increases the density or pore shrinkage [21]. The highest sintered density is achieved by the composites with the addition of silver (NCA) as a solid lubricant. This is due to the size of silver particles. The smaller size of silver particles was helpful during the mixing process in order for the formation of homogeneous structure [22]. Nano-sized silver was able to fill in the spaces between the main Ni-Cr particles inside the composite. This enhances the particle initial bonding, neck growth and pore rounding. Thus, greater pore shrinkage occurred and resulted in higher density compared to other composites. For self-lubricating composites, a certain level of porosity should be kept which is better for the self-lubrication mechanism to take place [23]. The porosity of ~20% is desirable for self-lubricating composites especially in bearings application [24]. Porosity will be explained further in the microstructure analysis section.

From Table 2, it is evident that the hardness of the composites is increasing as the sintering temperature increased. The highest hardness was achieved at the sintering temperature of 1200ºC for all composites. After sintering at 1200ºC, NC without any addition of solid lubricant achieved the highest hardness value. The results show that the addition of solid lubricant reduced the hardness of the composites, which is explainable by the fact that the additives possess higher ductility than the matrix. The addition of solid lubricant affects also the mechanical strength of the composites. The reduction of mechanical properties with the addition of solid lubricant is also supported by the previous research [25]. However, for the case of self-lubricating composites, it is important to have an adequate amount of solid lubricants added to the composites in order to have both excellent mechanical and tribological properties [26]. Based on the previous research, the adequate amount of solid lubricant added into the composite is in the range of 5-20wt% [26–28].

Microstructure of Ni-Cr based Composites

The effect of the sintering temperature on the microstructure and phase analysis of Ni-Cr based composites is shown in Figure 6-13. Figures 6-7 show the structure of sintered NC without the addition of solid lubricants after sintering at 1000ºC and 1200ºC, respectively. From the observation, the amount of porosity in Figure 6 is greater and the pore size is bigger compared to the porosity in Figure 7. The same is supported by the calculation of porosity which is reduced from 35% to 18%. At higher sintering temperature, the diffusion rate of Nickel into Chromium is higher, necking and bonding of particles are greater thus higher densification is expected. The XRD results in later sections would complement these results whereby the highest sintering temperature is limited to 1200ºC only.

Figures 8-9 show SEM and EDX analysis of NCM composite after sintering at 1200ºC respectively. From Figure 8 show the existence of small pores (black area). The MoS_2 addition is expected to fill in the voids at the time of mixing and also expected to incorporate better densification. The results are shown in Table 2 support this assumption. The diffusion of nickel particles into the Chromium and MoS_2 is better at a higher sintering temperature (1200ºC) shown in Figure 8. The existence of MoS_2 is supported in the EDX analysis in Figure 9. Based on calculations, the porosity was reduced from 32% to 17%, slightly better than pure Ni-Cr sample shown previously.

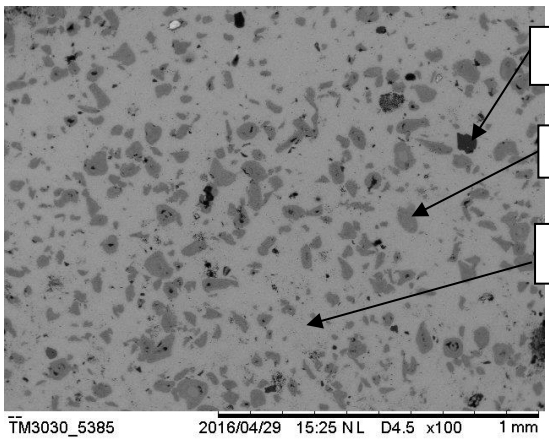


Figure 6. NC sintering at 1000ºC.

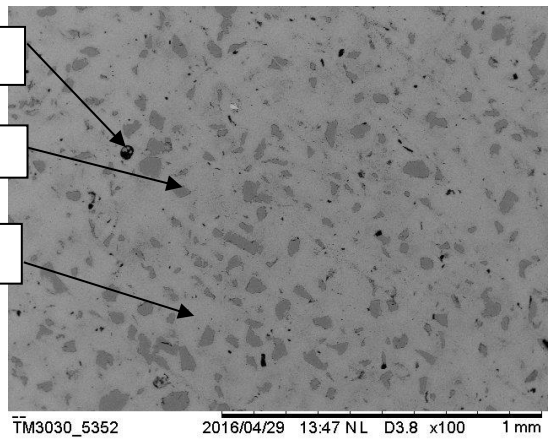


Figure 7. NC sintering at 1200ºC.

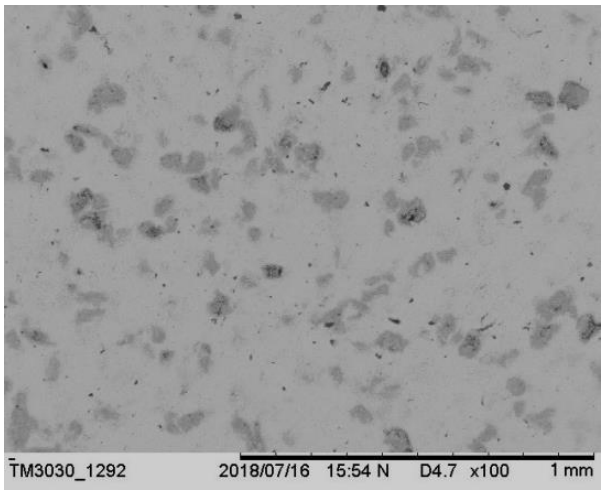


Figure 8. SEM micrograph of NCM composite.

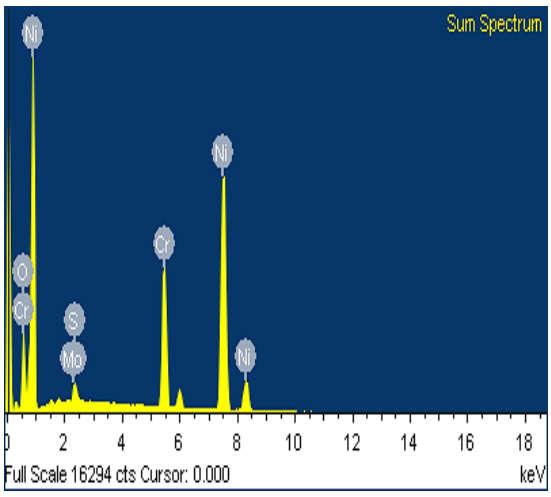


Figure 9. EDX for NCM composite.

Table 3. Elemental composition in NCM composite.

Element	O	S	Cr	Ni	Mo
Weight percentage (%)	8.68	0.42	18.84	76.42	2.49

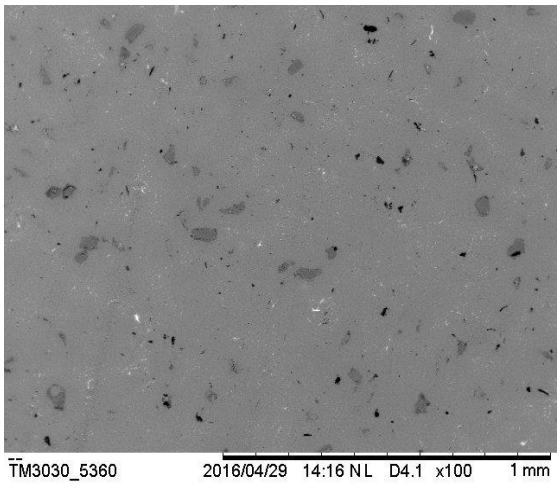


Figure 10. SEM micrograph of NCA composite.

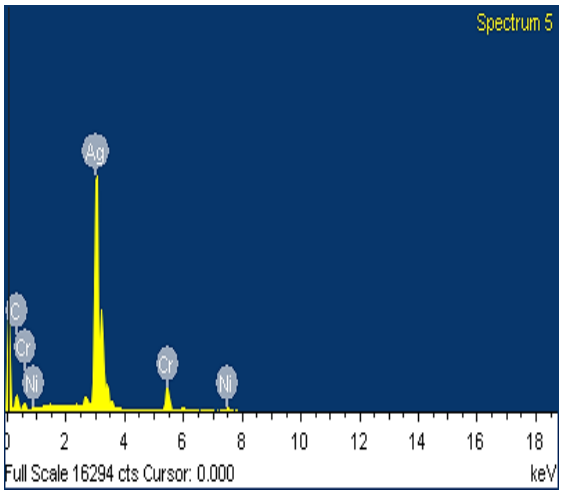


Figure 11. EDX of NCA composite on white phase.

Table 4. Elemental composition in NCA composite on white phase.

Element	C	Cr	Ni	Ag
Weight percentage (%)	3.71	8.62	1.76	85.91

Figure 10 and 11 represent the SEM and EDX analysis for NCA composite sintered at 1200ºC respectively. In the SEM micrograph of NCA composites, the white phases are detected and might represent Ag after the 5% addition into the composites. The EDX analysis confirmed the white phases as Ag as shown in Figure 11. From the SEM analysis, the black phases were decreased as a result of reduced in porosity from 30% to 15% as a result of better diffusion of Ni into Cr and Ag occurred at a higher sintering temperature.

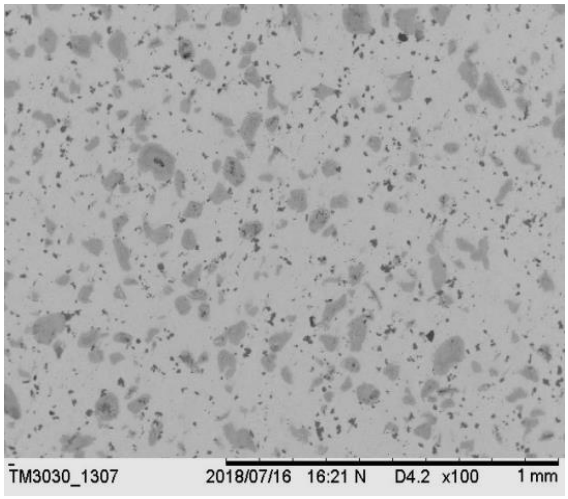


Figure 12. SEM micrograph of NCCf composite.

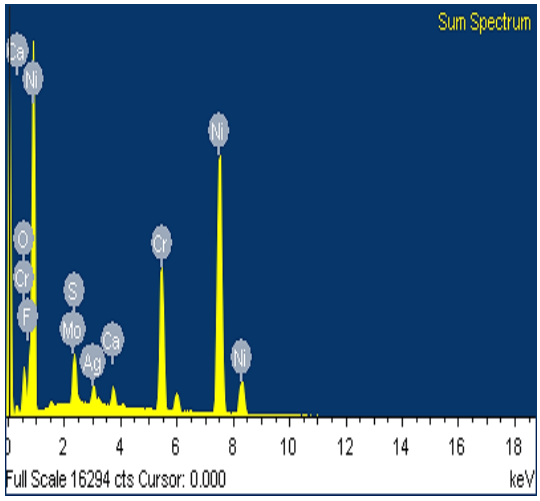


Figure 13. EDX of NCCf composite.

Table 5. Elemental composition in NCCf composite.

Element	O	F	Ca	Cr	Ni
Weight percentage (%)	4.05	3.20	1.50	17.12	74.13

Figure 12 and 13 show SEM and EDX analysis of NCCf composite after sintering at 1200ºC, respectively. The presence of CaF_2 is detected from the EDX analysis in Figure 13. It was observed that the Cr grains get reduced in size when sintering at higher temperatures. Thus, the diffusion occurred rapidly at a higher temperature which is 1200ºC and formed good interface bonding of Ni-Cr and Ni-Cr-solid lubricants (MoS_2, Ag and CaF_2). This maybe a result of partial oxidation at higher temperature, which would be discussed at a later stage. Based on calculations, the porosity was reduced from 29% to 13%. XRD analysis is conducted before and after sintering at 1200ºC and the effect of sintering on the phase analysis of Ni-Cr composites are shown in Figure 14-15.

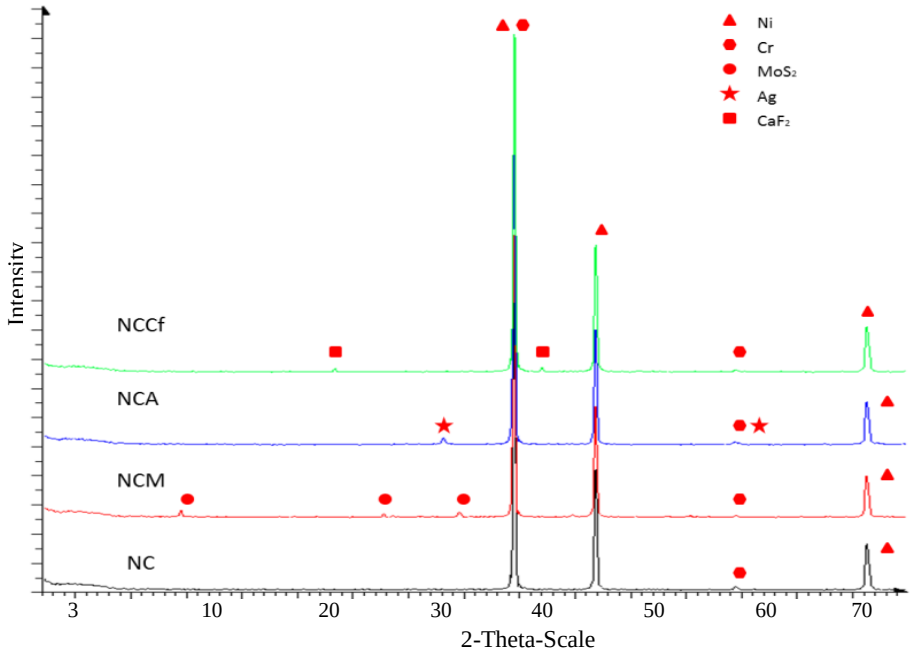


Figure 14. XRD analysis of Ni-Cr composites before sintering.

Figures 14-15 show the XRD analysis of Ni-Cr composites before and after sintering respectively. The analysis is done using the EVA software and confirmed with the JSPDS cards. In Figure 14, the peaks of solid lubricant (MoS_2, Ag and CaF_2) are detected to confirm the existence of solid lubricants before the sintering. After ball milling, the intensities of the peaks become weak due to the refinement of grain size during milling. Figure 15 shows the peak of Ni and Cr for all composites (NC, NCM, NCA and NCCf) reduced its intensities and diminished after 1 hour of sintering due to the amorphization and internal stresses in these phases [30].

In Figure 15, the second Cr peak is visible for the pure Ni-Cr alloy after sintering for NC composite. This means Cr is present, although it may be camouflaged with oxide layer on its surface. The oxidation of chromium is also evident when sintering at 1200°C. Therefore, there are additional peaks formed which represent the formation of Cr_2O_3. Besides that, NCM composite also shows the addition of new peaks which represent Cr_2O_3. The formation of Cr_2O_3 was expected due to the existence of a small amount of oxygen in the powder acquired during the milling process, especially when sintered at 1200°C. However, the formation of Cr_2O_3 may become anti wear agent [31] and also a high temperature solid lubricant [32]. Few researchers have also used Cr_2O_3 as one of the components in self-lubricating composites as it offers good lubricity up to 1000°C [33].

For NCA composite, the intensities of Ag at 2 Theta angle of 38 was slightly increased after the sintering process as seen in Figure 12. This may be because of dissolved or embedded Ag may have precipitated out and combined to form larger grains [34]. The microstructure in Figure 10 also shows the homogenous distribution of silver in Ni-Cr based composites and slight increase in grain size compared to original powder particles, which attributed to the highest sintered density compared to other composites.

Therefore, NCA composite has produced the best properties of hardness and sintered density. Though the hardness of all composites with the addition of solid lubricant is close, NCA composite has shown maximum sintered density. The existence of Ag even after sintering is supported from the XRD analysis in Figure 15. Further characterization of surfaces and study of friction coefficient would reveal which combination of lubricants provides better results.

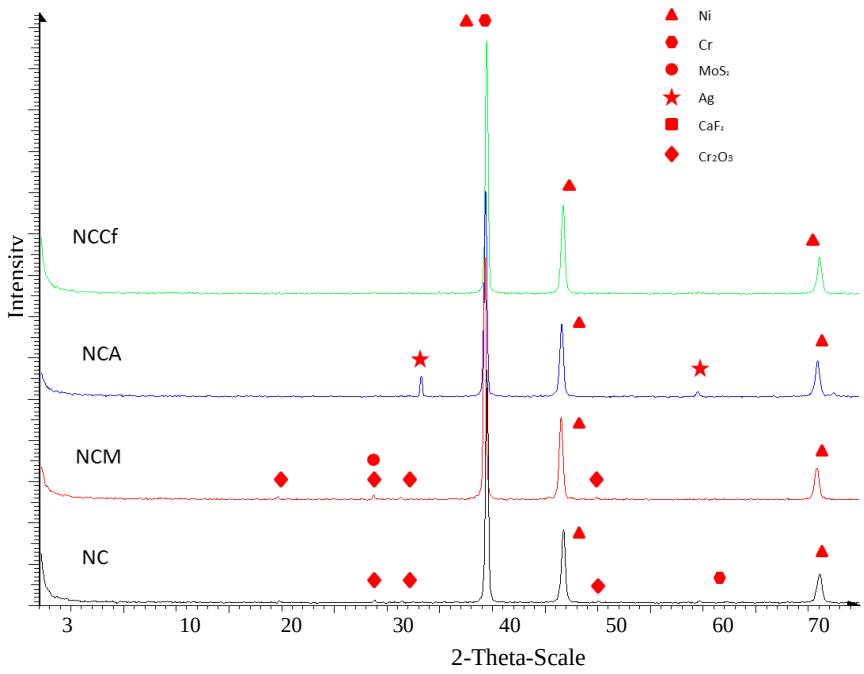


Figure 15. XRD analysis of Ni-Cr composites after sintering.

CONCLUSIONS

The effect of sintering temperature on pure Ni-Cr composites and with the single addition of solid lubricants (MoS_2, Ag and CaF_2) was studied. The sintered density, relative density and hardness of the Ni-Cr composites were increased as sintering temperature increased from 1000 to 1200°C. Maximum relative density was achieved at the sintering temperature of 1200°C for all types of composites. NCA composites exhibited the highest relative density at a sintering temperature of 1200°C. The SEM microstructure proved that the fine size of the Ag phase was distributed homogeneously, contributed to filling the pores and thus increased the densification of the composite. In all cases, the achieved density was around 85% or more of the theoretical density.

Optimization of sintering temperature up to 1200°C resulted in achieving maximum relative density and high hardness of all the composites. Even after sintering at 1200°C, the presence of solid lubricants still exists as supported either by SEM, EDX and XRD analysis. The limitation of sintering temperature up 1200°C as minimum porosity (less than 20%) is achieved for good self-lubrication requirements. Future work is to concentrate on more than one solid lubricants in the Ni-Cr composites for wide range temperature applications.

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