Sintered Fe-Ni-Si-C alloys

Tiwat Kaewkam¹, Panya Kansuwan¹, Naoto Ohtake², Pongsak Wila³, Rungtip Krataithong³, Nattaya Tosangthum³, Thanyaporn Yotkaew⁴*, and Ruangdaj Tongsri³

¹Department of Mechanical Engineering, Faculty of Engineering, King Mongkut’s Institute of Technology Ladkrabang (KMITL), Bangkok Thailand 10520
²Department of Mechanical Engineering, Major in Engineering Sciences and Design, Tokyo Institute of Technology, Tokyo Japan 152-8550
³Particulate Materials Processing Technology (PMPT), Metal and Manufacturing Processes Research Group, National Metal and Materials Technology Center, 114 Paholyothin Road, Khlong Nueng, Klong Luang, Pathum Thani Thailand 12120

* Corresponding Author: thanyy@mtec.or.th

Abstract
The aim of this research is to explore the effect of nickel content on microstructural development and mechanical properties of sintered Fe-Ni-Si-C alloys. The sintered alloys were prepared from powder mixtures of pre-alloyed Fe-Ni-based powders (nickel contents varied as 0.45, 0.90, 1.80, and 4.00 wt.%) and fixed 4 wt.% silicon carbide powder by using a powder metallurgical process. Sintering was performed in a vacuum furnace at 1250°C for 45 minutes and slow cooling in the furnace. Microstructures of sintered alloys varied with nickel contents. Nickel showed a strong influence on promoting bainitic ferrite/martensite-austenite (BF/M-A) structure formation. Retained austenite in BF/M-A structure was found to increase with increasing nickel content. Tensile properties (strength and elongation value) of sintered alloys increased with increasing nickel content.

Keywords: Sintering, Fe-Ni-Si-C alloys, Ferrite, Martensite-austenite, Mechanical property.

1. Introduction
Thailand has a geographic location suitable to become a primary regional economic hub. Not only in trade, investment, and tourism but also in communication and transportation networks, that connect to other regions around the world. The Thai government has, in recent years, made investment in domestic infrastructure, a top priority. Railway transportation is increasingly important as a mass transit, to link destinations with manageable travelling time. With the project of high-speed railway development, sintered friction materials (SFMs) are expected to be widely used. These materials are used in train brake system and have many advantages, such as high performance, wear resistance, improved fade resistance, enhanced wet-effectiveness, and extended life SFMs all important components of rail brakes to ensure safety and comfort expectation [1]. SFMs are composites commonly consisting of graphite particles embedded in metallic matrix [2], [3]. The graphite particles act as solid lubricant for anti-wear purpose. According to the recent study [4], the SFM matrix has important role on braking performance such as...
stable coefficient of friction (COF) and wear rate. This means the composite matrix integrity is important for high-speed train SFM brake pads. Previous works have shown that sintered composites having graphite particles embedded in iron-base matrices could be produced by using the traditional ‘press and sinter process’ [5–8]. These sintered composites have different matrices depending on the compositions of iron-base powders employed. The matrix microstructure has prime contribution to mechanical properties of a sintered composites.

Research works have shown that nickel addition to steels is able to modify microstructure hence improve mechanical property. The increase in Ni contents (0-6%) causes microstructural change from dominant granular bainite to lath bainite and/or lath martensite [9]. The roles of nickel addition include (i) lowering the pearlite start (Ps) and the bainite start (Bs) temperatures and shifting both pearlitics, (ii) and bainite transformation curves to the right-hand side of continuous cooling transformation (CCT) diagram and (iii) retardation of isothermal bainite transformation kinetics by shift the bainite transformation curve to the right-hand side of time-temperature-transformation (TTT) diagram [10]. The decrease of Bs and martensite start (Ms) temperature with nickel addition [11] or increasing nickel content [12] were previously reported.

The shifting of the pearlite transformation curve to the right-hand side of CCT diagram opens the opportunity for a steel produced under a considerably slow cooling rate to avoid pearlite transformation at a high temperature thus undergoes other lower temperature phase transformations. The additions of 1-3 wt.% nickel powder to sintered Fe-Cr-Mo base steels, processed under a slow or furnace cooling, seem to prove that concept [13]. The retardation of isothermal bainite transformation kinetics due to nickel addition is reported in [10, 14].

Due to the important roles of nickel addition as given above, it was hypothesized that by using pre-alloyed nickel-containing powders, the matrices of sintered SFMs would be developed under the influence of nickel content. Due to the delay of pearlite transformation, other transformations occurring below the pearlite field are expected. One of low temperature transformation products of ductile cast irons known as ausferrite consisting of ferrite and carbon-rich austenite is expected to form by the nickel influence in sintered nickel-containing SFMs. The ausferrite is one type of microstructural features important for mechanical property improvement in cast ductile irons [15–19]. The effects of microstructural components, varied with nickel content, on mechanical properties of sintered nickel-containing SFMs had been investigated in this work.

2. Materials and Methods

2.1 Materials preparation

The sintered alloys were prepared from powder mixtures of pre-alloyed Fe-Ni based powders (nickel contents varied as 0.45, 0.90, 1.80, and 4.00 wt.%) and fixed 4 wt.% silicon carbide powder (shown in Table 1) by using a powder metallurgical process. The powder mixtures were compacted into tensile test bars with a green density of 6.50 ± 0.05 g/cm3. For each steel alloys, sintering of green compacts were performed in a vacuum furnace at 1250°C for 45 minutes and slowly cooled in the furnace. The density of green and sintered specimens was determined in accordance with MPIF Standard 10, ASTM B783.

| Alloy | Pre-alloyed powder | SiC (wt.%) | Nominal composition (wt.%) |
|------|--------------------|------------|---------------------------|
|      |                    |            | C | Si | Cr | Ni | Mo | Mn | Fe |
| 0.45Ni | Fe-0.45Ni-0.60Mo-0.15Mn | 4.0 | 1.2 | 2.8 | 0.0 | 0.45 | 0.85 | 0.15 | Bal. |
| 0.90Ni | Fe-0.90Ni-1.00Mo-0.45Cr-0.45Mn | 4.0 | 1.2 | 2.8 | 0.45 | 0.90 | 1.00 | 0.45 | Bal. |
| 1.80Ni | Fe-1.80Ni-0.55Mo-0.20Mn | 4.0 | 1.2 | 2.8 | 0.0 | 1.80 | 0.55 | 0.20 | Bal. |
| 4.00Ni | Fe-4.00Ni-0.50Mo-0.20Mn | 4.0 | 1.2 | 2.8 | 0.0 | 4.00 | 0.50 | 0.20 | Bal. |
2.2 Microstructure investigation
Specimens for metallographic examination were ground on silicon carbide papers from 80 to 4000 grits and then progressively polished with 6, 3, and 1μm diamond pastes, respectively. The polished samples were etched with 2% Nital. Microstructures of the sintered specimens were observed by using optical microscopy (OM) and scanning electron microscopy (SEM) equipped with an EDS.

2.3 Hardness and tensile test
Rockwell scale B hardness test (HRB) was carried out on un-etched specimens using 100 kgf loads with 15s indentation time (Standard ISO 6508-1). The mean values were based on ten different areas. The tensile property of the sintered materials was carried out at room temperature using Instron Universal Instrument with speed 1 mm/min. Three samples were tested for each sintered steel and the average value was reported.

3. Results and discussion
3.1 Microstructure and phase characterization
Sintered Fe-Ni-Si-C alloys showed microstructures resembling those of ductile cast irons (Figure 1). The sintered 0.45Ni (Figure 1a) and 1.80Ni (Figure 1c) alloys showed common microstructural feature consisting of black particle surrounded with ferrite and striated structure, whereas sintered 0.90Ni (Figure 1b) and 4.00Ni (Figure 1d) had black particle surrounded with only striated structure. Microstructural observation using SEM revealed that the striated structure in the sintered 0.45Ni alloy (Figure 2a) consisted of pearlite and bainitic ferrite/martensite-austenite (BF/M-A) with higher proportion of pearlite. The striated structure in the sintered 0.90Ni alloy (Figure 2b) consisted of BF/M-A structure. The striated structure in the sintered 1.80Ni alloy (Figure 2c) consisted of BF/M-A and pearlite with lower proportion of pearlite. The matrix of the sintered 4.00Ni alloy (Fig. 2d) consisted of BF/M-A and martensite with lower proportion of martensite. The XRD patterns of the sintered Fe-Ni-Si-C alloys as given in Figure 3 show peaks corresponding to two different crystal structures, i.e., body-centered cubic (bcc) crystal structure belonging to ferrite (α) phase and face-centered cubic (fcc) crystal structure belonging to austenite (γ) phase.

The formation of microstructure consisting of black particle surrounded by ferrite and striated structure in sintered Fe-Ni-Si-C alloys is already explained and given in [6]. However, the striated patterns in the sintered nickel-containing alloys are quite different from those observed in sintered Fe-Ni-Si-C alloys. Thus, additional discussion in this work is focused on the formation of striated structure. When the striated structures in sintered 0.45Ni alloy (Figure 2a), 1.80Ni alloy (Figure 2c) and 4.00Ni alloy (Figure 2d) are compared, the effect of nickel content on phase transformations is remarkable. The proportion of pearlite decreases but that of BF/M-A structure increases with increasing nickel content. Taking the sintered Fe-Mo-Si-C alloys [6,7] as references, all sintered nickel-containing alloys (in this work) have higher proportions of BF/M-A structure. The reduction or disappearance of ferrite and pearlite in sintered nickel-containing alloys confirms the effect of nickel on shifting of high-temperature transformation curves to the right-hand side of CCT diagram [10]. Experimental results suggest that the higher nickel content the farther high-temperature transformation curves are shifted and the wider window is opened for the low-temperature transformation products (BF/M-A structure and martensite) to form.

The formation of low-temperature transformation products dictates the whole matrices of the sintered 4.00Ni alloy (Figure 2b) and 0.90Ni alloy (Figure 2d). In the former alloy, the low-temperature transformations are clearly influenced by nickel. In the latter one, nickel concentration alone cannot totally suppress high-temperature transformations. In this alloy, the synergistic effects on high-temperature transformation suppression are supposed to obtain from chromium (1.0 wt.%) and molybdenum (1.0 wt.%). The strong and medium effects of molybdenum and chromium, respectively, in suppressing pearlite formation were previously reported [20]. Taking the overall microstructures of these two alloys into consideration, their microstructures are similar to those of austempered ductile
irons. The microstructure of sintered 0.90Ni and 4.00Ni alloys consist of black particle (graphite-containing particle according to \([6–8]\)) surrounded with the BF/M-A structure matrix while that of austempered ductile irons consist of graphite particle surrounded with auferrite (ferrite and carbon-rich austenite) matrix \([5–19]\).

![Image of optical microstructures of sintered Fe-Ni-Si-C alloys](image1)

**Figure 1.** Optical microstructures of sintered Fe-Ni-Si-C alloys.

![Image of SEM microstructures of sintered Fe-Ni-Si-C alloys](image2)

**Figure 2.** SEM microstructures of sintered Fe-Ni-Si-C alloys.
3.2 Mechanical property
The sintered Fe-Ni-Si-C alloys showed appreciably high tensile strengths (ultimate tensile and yield strengths) as given in Figure 4. Ductility values were quite low, no higher than 3.0%. Both strength and elongation values increased with increasing nickel content. Rockwell scale B hardness value showed relationship with nickel content for most sintered alloys, except the value drop in the sintered 1.80Ni alloy. Note that the prominent strengths and hardness of the sintered 0.90Ni alloy is also attributed to high Cr and Mo contents.

![Figure 3. XRD of sintered Fe-Ni-Si-C alloys.](image)

![Figure 4. Mechanical properties of sintered Fe-Ni-Si-C alloys, according to nickel content.](image)

The increase of tensile strength is attributed to microstructural components. Experimental results (Figure 2) provide the evidences of low-temperature transformation products, such as BF/M-A and martensite, as the root causes of alloy strengthening. Particularly, the sintered 4.00Ni alloy having tensile
strength over 1 GPa has matrix consisting mostly of BF/M-A and martensite. The elongation value increases with increasing nickel content (Figure 4c) but is not directly related to retained austenite volume indicated by the $\gamma$ (111) peak intensities shown by XRD patterns in Figure 3. The ductility of the sintered alloys is partly influenced by retained austenite. The hardness (HRB) value has no direct relationship with nickel content (Figure 4d). The hardness value drops in the sintered 1.80Ni alloy is attributed to pearlite structures distributed in BF/M-A matrix.

3.3 Fracture surface
Fracture surface of the sintered 0.45Ni alloy with the lowest elongation value showed cleavage with river pattern (Figure 5a) whereas that of the sintered 4.00Ni alloy with higher elongation value showed mixture of cleavage (on the right) and quasi-cleavage (on the left) (Figure 5b). Cleavage, as a typical sign of brittle fracture, found in pearlite-containing matrix of sintered 0.45Ni alloy, may not be uncommon as it was found on a fracture surface of a fully pearlitic eutectoid steel [21,22]. Quasi-cleavage, as one form of cleavage, exhibits both cleavage and plastic deformation [23]. This fracture mode found in the sintered 4.00Ni alloy reflects small plastic deformation as this alloy has high strength but small elongation value.

Figure 5. Fracture surfaces of sintered Fe-Ni-Si-C alloys.

4. Conclusion
Sintered Fe-Ni-Si-C alloys, designed and fabricated for application as SFMs, could be produced from mixtures of pre-alloyed Fe-Ni and fixed 4 wt.% silicon carbide powders by using traditional press and sinter process. Nickel content in pre-alloyed showed influence on microstructures and mechanical properties of sintered alloys. High nickel content could suppress ferrite and pearlite transformations and open a window for low-temperature transformations of BF/M-A and martensite. The low-temperature transformation products showed strong influence on mechanical properties of sintered alloys.

Acknowledgments
This work is funded by the project P1951261 under National Science and Technology Development Agency (NSTDA). The first author is also financially supported by TAIST-Tokyo Tech scholarship. The authors are grateful for supports from Thailand National Metal and Materials Technology Center (MTEC) and King Mongkut’s Institute of Technology Ladkrabung (KMITL). The authors are also grateful for the material for this research, courtesy from Rio Tinto Metal Powders of Canada (via P.S. Steel Co., Ltd., of Thailand and Rio Tinto Iron & Titanium (Suzhou) Co., Ltd., of China).

References
[1] Günay M Korkmaz M E and Özmen R 2020 Eng. Sci. Technol. an Int. J. 23 421-431
[2] Xiao J K Xiao S X Chen J and Zhang C 2020 Tribol. Int. 150 106357
[3] Pérez B and Echeberria J 2019 Influence of abrasives and graphite on processing and properties of sintered metallic friction materials Heliyon 5(8) E02311
[4] Peng T Yan Q and Zhang X 2018 Tribol. Lett. 66 1-13
[5] Chakthin S Poolthong N and Tongsri R 2008 Adv. Mater. Res. 55-57 357-360
[6] Ruangchai K Wiengmoon A Morakotjinda M Krataitong R Tanprayoon D Yotkaew T Tosangthum N Patakham U and Tongsri R 2017 *Key Eng. Mater.* **751** 47-52
[7] Ruangchai K Wiengmoon A Morakotjinda M Tosangthum N and Tongsri R 2018 *J. Phys. Conf. Ser.* **1144** 012099
[8] Ruangchai K Wiengmoon A, Krataitong R Yotkaew T Tosangthum N and Tongsri R 2018 *J. Phys. Conf. Ser.* **1144** 012147
[9] Mao G Cao R Yang J Jiang Y Wang S Guo X Yuan J Zhang X and Chen J 2017 *J. Mater. Eng. Perform.* **26** 2057-2071
[10] Tian J Xu G Jiang Z Hu H and Zhou M 2018 *Met. Mater. Int.* **24** 1202-1212
[11] Kong J and Xie C 2006 *Mater. Des.* **27** 1169-1173
[12] Yang H S and Bhadeshia H K D H 2008 *Mater. Sci. Technol.* **24** 335-342
[13] Tosangthum N Morakotjinda M Krataitong R Wila P Yodkaew T Vetayanugul B Boontetim W and Tongsri R 2018 *Mater. Today Proc.* **5** 9351-9358
[14] Yao Z Xu G Hu H Yuan Q Tian J and Zhou M 2019 *Trans. Indian Inst. Met.* **72** 1167-1174
[15] Zimba J Simbi D J and Navara E 2003 *Cem. Concr. Compos.* **25** 643-649
[16] Eric O Sidjanin L Miskovic Z Zec S and Jovanovic M T 2004 *Mater. Lett.* **58** 2707-2711
[17] Erić O Rajnović D Zec S Sidjanin L and Jovanović M T 2006 *Mater. Charact.* **57** 211-217
[18] Ovali I Kilicli V and Erdogan M 2013 *ISIJ Int.* **53** 375-381
[19] Kaczorowski M and Krzyńska A 2007 *Arch. Foundry Eng.* **7** 161-166
[20] Park Y J and Fletcher F B 1986 *J. Heat Treat.* **4** 247-252
[21] Bernstein I M and Alexander D J 1989 *Metall. Trans. a* **20** 2321-2335
[22] Lewandowski J J and Thompson A W 1986 *Metall. Trans. A* **17** 1769-1786
[23] Tartaglia J M Lazzari K A Hui G P and Hayrynen K L 2008 *Metall. Mater. Trans. A Phys. Metall. Mater. Sci.* **39 A** 559-576