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IMPROVEMENT OF STRENGTH AND OXIDATION RESISTANCE AT HIGH TEMPERATURE IN AISI 4140 STEEL BY MICRO-ALLOYING CHROMIUM AND TUNGSTEN FOR AUTOMOTIVE ENGINE APPLICATIONS

Increasing the operating temperature and pressure of an automotive engine and reducing its weight can improve fuel efficiency and lower carbon dioxide emissions. These can be achieved by changing the engine piston material from conventional aluminum alloy to high-strength heat-resistant steel. American Iron and Steel Institute 4140 modified steels (AISI 4140 Mod.s), which have improved strength, oxidation resistance, and wear resistance at high temperature were developed by adjusting the AISI 4140 alloy compositions and optimizing the heat treatment process for automotive engine applications. In this study, the effects of modifying alloy compositions on the microstructure, mechanical properties (both at room and high temperatures), and oxidation of AISI 4140 Mod.s were investigated. Effective grain refinement occurred due to the influence of high-temperature stable carbide forming elements such as Mo, and V. The bainite structure changed to martensite structure under the influence of Cr and Ni. As the Cr and W contents increased, the oxidation resistance was improved, and the oxide layer thickness decreased after 10 hours exposure at 500°C. The AISI 4140 Mod. exhibited a 35% improvement in room temperature strength, 70% improvement in high-temperature strength, and 40% improvement in high-temperature oxidation resistance compared to conventional AISI 4140.

Keywords: AISI 4140; micro-alloying; microstructure; mechanical property; heat-treatment

1. Introduction

American Iron and Steel Institute 4140 (AISI 4140) is a Cr-Mo low-alloy steel with high strength, toughness, and corrosion resistance. Due to affordable price, it has been widely applied in various industries such as automobile and oil & gas, etc. [1,2]. To improve fuel efficiency and reduce carbon dioxide emissions of automobile, researches have been conducted to reduce vehicle weight and improve engine efficiency [1,2]. The automotive engines efficiency can be enhanced by increasing the operating temperature and pressure. Aluminum alloys have been conventional automotive engine materials, however face application temperature limitation. Replacing aluminum alloys with heat-resistant high-strength steel in engines has the advantages of increasing operating temperature and reducing engine volume. To apply AISI 4140 steel to engines, high-temperature strength, oxidation resistance and wear resistance are needed to be improved [3]. In this study, three AISI 4140 modified steels (AISI 4140 Mod.s) were manufactured by adding elements that enhance high-temperature properties. The microstructure and

mechanical properties of modified steels were compared with those of conventional AISI 4140.

2. Experimental

In this study, three types (P1~P3) of AISI 4140 Mod. forged round rods were manufactured by modifying alloy elements of AISI 4140 steel (P0), as shown in Table 1. To increase strength, oxidation resistance and wear resistance at engine operation temperature of 500°C, high temperature stable carbide forming element of Cr, Mo, V and W were added. Ni can increase hardenability and induce fine martensitic structure with high strength and toughness simultaneously. Mo, Ni, and V were added equally to all alloys. Regarding Cr and W, P1 was manufactured by adding 3 wt.% Cr, and P2 by adding 4 wt.% Cr. P3 was made of 3 wt.% Cr and 1 wt.% W. The materials were austenitized at 880°C for 1 hour, followed by water quenching, and tempered at 580°C for 1.5 hours. Microstructure analysis was conducted using an Field Emission Scanning Electron

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Microscope (FE-SEM; Hitachi S-4800). Tensile tests (ASTM E5) were performed at room temperature and at high temperature (500°C), using a 100 kN MTS Landmark universal testing machine at a cross head speed of 1.44 mm/min. The dimensions of the cylindrical tensile specimens were 24 mm (gage length) and 6 mm (diameter). Charpy impact tests were performed at room temperature using an automatic ZWICK PSW750 impact test machine. Charpy impact specimens were prepared according to ASTM E23 (10 mm × 10 mm × 55 mm, 45° 2 mm V-notch). High temperature oxidation tests were performed at 500°C for 10 hours in an air environment box furnace. Oxidation test specimen dimensions were 10 mm (diameter) × 5 mm (length) and polished until 600 grit. The oxidation rate was measured by the weight gain after oxidation.

3. Results and discussion

The microstructures of the AISI 4140 (P0) and three types of AISI 4140 Mod.s (P1, P2, P3) are shown in Fig. 1. P0 showed a mixture of acicular ferrite and bainite, while P1, P2, and P3

exhibited tempered martensite. The block thickness of AISI 4140 Mod.s decreased from about 0.6 μm for P0 to 0.2 μm for P1~P3. The block acts as effective grain in both bainite and martensite, thereby causing grain refinement effects. In AISI 4140 Mod.s, the addition of Mo, V, Nb, and W generated carbides with high temperature stability, which delayed grain growth by pinning the prior austenite boundaries during austenitizing heat treatment [4,5]. Grain refinement leads to an increase in room temperature strength, room temperature toughness, and high temperature strength. Additionally, the presence of high-temperature stable carbides retards softening from exposure to high-temperatures during piston operation [5]. The incorporation of micro-alloys such as Cr, Mn and Ni improves hardenability, and the induced martensite offers advantages in terms of mechanical properties (strength, toughness, hardness, and homogeneity) [1,3,4,6,7].

High-temperature oxidation resistance can be improved by adding Cr, W, Al, or Si; however, the material toughness decreases significantly with the addition of Al and Si [7-9]. Therefore, Cr and W were selected for enhancing the oxidation resistance.

Fig. 2 shows the mechanical properties. P1, P2, and P3 showed increased strength and hardness at room temperature

TABLE 1

Chemical compositions of AISI 4140 (P0) and AISI 4140 Mod.s (P1, P2, P3)

Element (wt.%)	C	Si	Mo	Cr	Ni	Mn	Cu	V	W	Fe
P0 (AISI 4140, reference)	0.42	0.27	0.14	1.04	0.08	0.88	0.17	0.01	—	Bal.
P1 (3% Cr add)	0.39	0.32	1.11	3.02	0.52	0.97	0.21	0.54	—	Bal.
P2 (4% Cr add)	0.40	0.35	1.15	3.89	0.53	1.00	0.23	0.56	—	Bal.
P3 (3% Cr & 1% W add)	0.34	0.30	1.09	3.05	0.52	0.95	0.21	0.54	0.93	Bal.

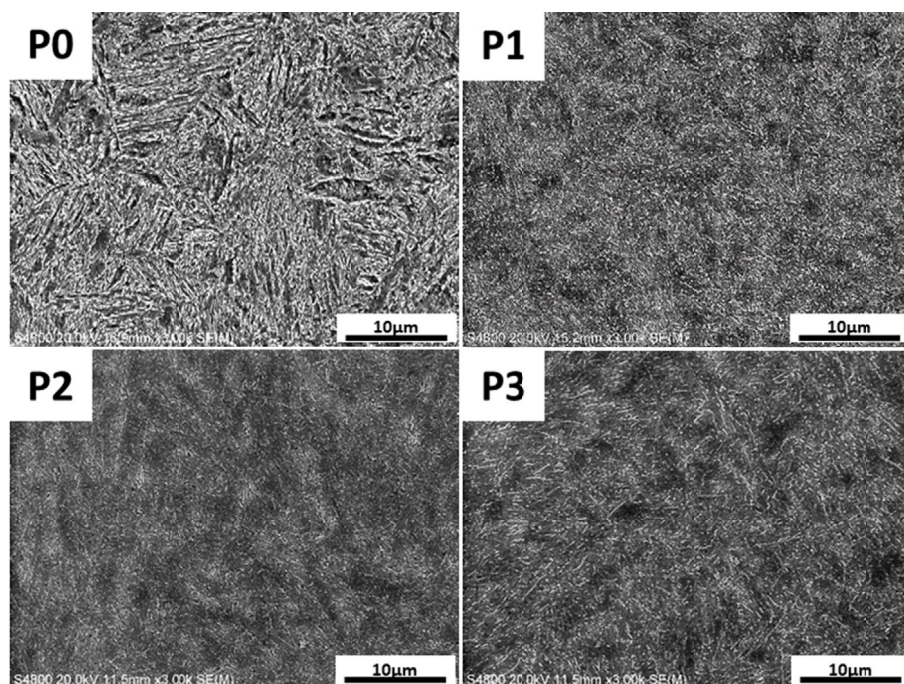


Fig. 1. Microstructure of P0, P1, P2, and P3 using FE-SEM

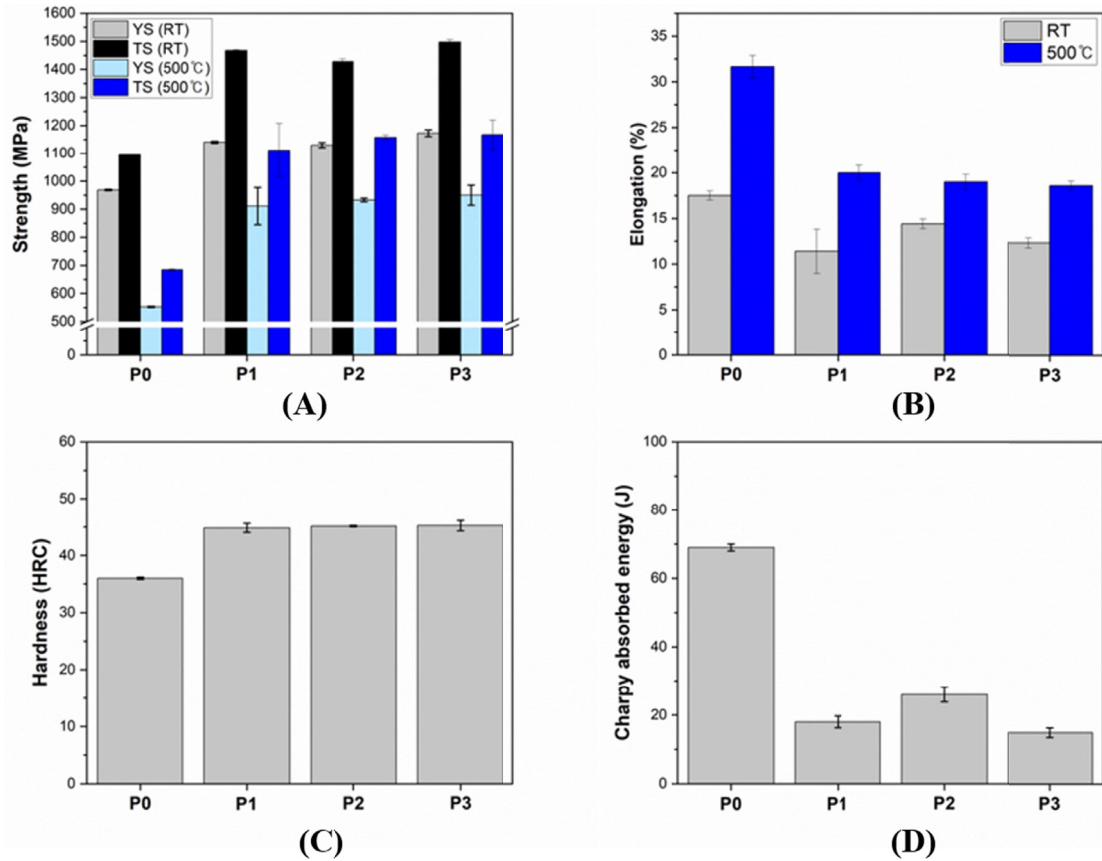


Fig. 2. Mechanical properties of P0, P1, P2, and P3: (a) strength at room temperature and 500°C, (b) elongation at room temperature and 500°C, (c) hardness at room temperature, (d) toughness at room temperature

compared to P0 (Fig. 2a). This increase was attributed to precipitation hardening and grain size refinement caused by elements Cr, Mo, W, and V [4]. Additionally, a martensitic hardening occurred due to improved hardenability resulting from the increased total amount of alloy including Ni [4]. The behaviors were consistent with the microstructural analysis. Although the toughness decreased (Fig. 2d), the elongations of 12% or more, which was the requirement for diesel engine piston, was satisfied (Fig. 2b).

At 500°C, the tensile strength tended to decrease by 300 MPa in P1~P3 and 400 MPa in P0 compared to the tensile strength at room temperature. The strength decrease in P1 to P3 was smaller than that in P0, which was interpreted as the effect of Mo, V, and Nb forming stable carbide at high temperatures. Compared to P1, both P2 and P3 had an increased Cr content by 1 wt.% and W content by 1 wt.%, respectively. However, the high-temperature mechanical properties were similarly measured, and the effect of these elements on high-temperature mechanical properties was not obvious [7, 8]. While W is a high-temperature stable carbide-forming element, the amount of carbide formed per unit weight by W was less than that of other elements due to its large atomic weight [10]. As a result, W had a smaller strengthening effect by weight. High-temperature oxidation resistance tests were conducted to confirm the effect of increasing the Cr and W content [11].

Hardness showed an increased value in P1~P3 compared to P0 (Fig. 2c), although the increasing ratios were lower than

those of the tensile strength. Impact toughness of P2, which had the highest Cr content among AISI 4140 Mod.s, exhibited the highest value; however, it was not very high compared to the other materials (Fig. 2d).

Fig. 3a shows the result of measuring the weight gain per unit area after 10 hours of exposure to air at 500°C. P0 exhibited the largest weight gain, and the weight gain tended to decrease in the order of P1, P2, and P3. Weight gain occurs when material elements combine with oxygen in the air at high temperature. The smaller the weight gain, the less oxidation occurred, and the higher corrosion resistance exhibited. Oxidation resistance increased as the Cr content increased to 0.14, 3.02, and 3.89 wt.% in P0, P1, and P2 respectively. P3 with 1 wt.% of W added, showed the weight gain similar to that of P2 with 1 wt.% of Cr added, and W exhibited an oxidation resistance effect similar to that of Cr in AISI 4140 Mod.s.

Fig. 3b and Fig. 3b. 4 show the thickness and composition distribution of the oxide layer. The oxide layer consisted of an iron oxide layer on the surface and a Cr-rich oxide layer on the inside (Fig. 4). The total oxide layer thickness became thinner in the order of P0, P1, P2, and P3, which was consistent with the weight gain results. The thicknesses of the Cr oxide layer were similar, at about 1.5 μm for all materials, while the thickness of iron oxide layer decreased with increasing Cr + W content.

Cr is a more oxidizing element than iron and forms a Cr_2O_3 oxide layer on the surface of AISI 4140 in high-temperature air.

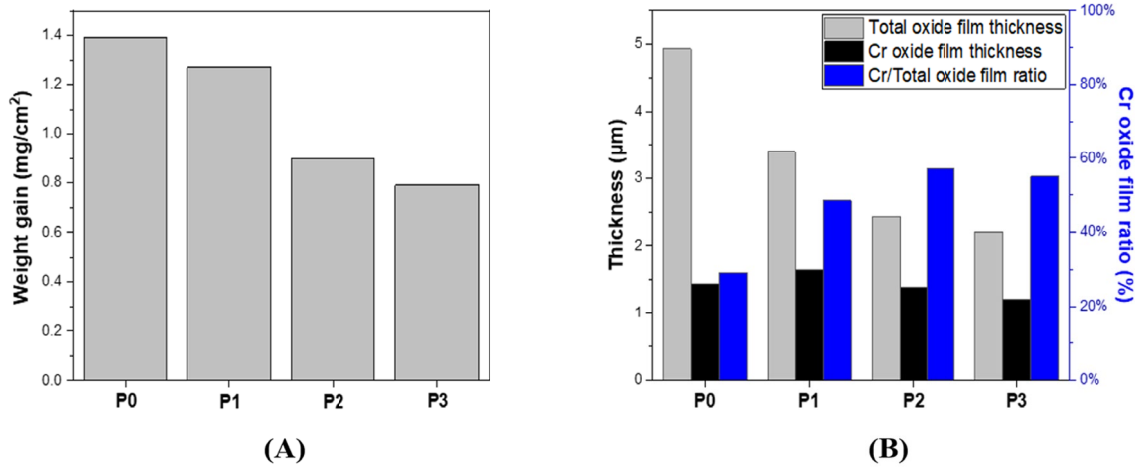


Fig. 3. High temperature oxidation resistance tests of P0, P1, P2, and P3 at 500°C air environments; (a) weight gain, (b) Thickness details of oxide layer

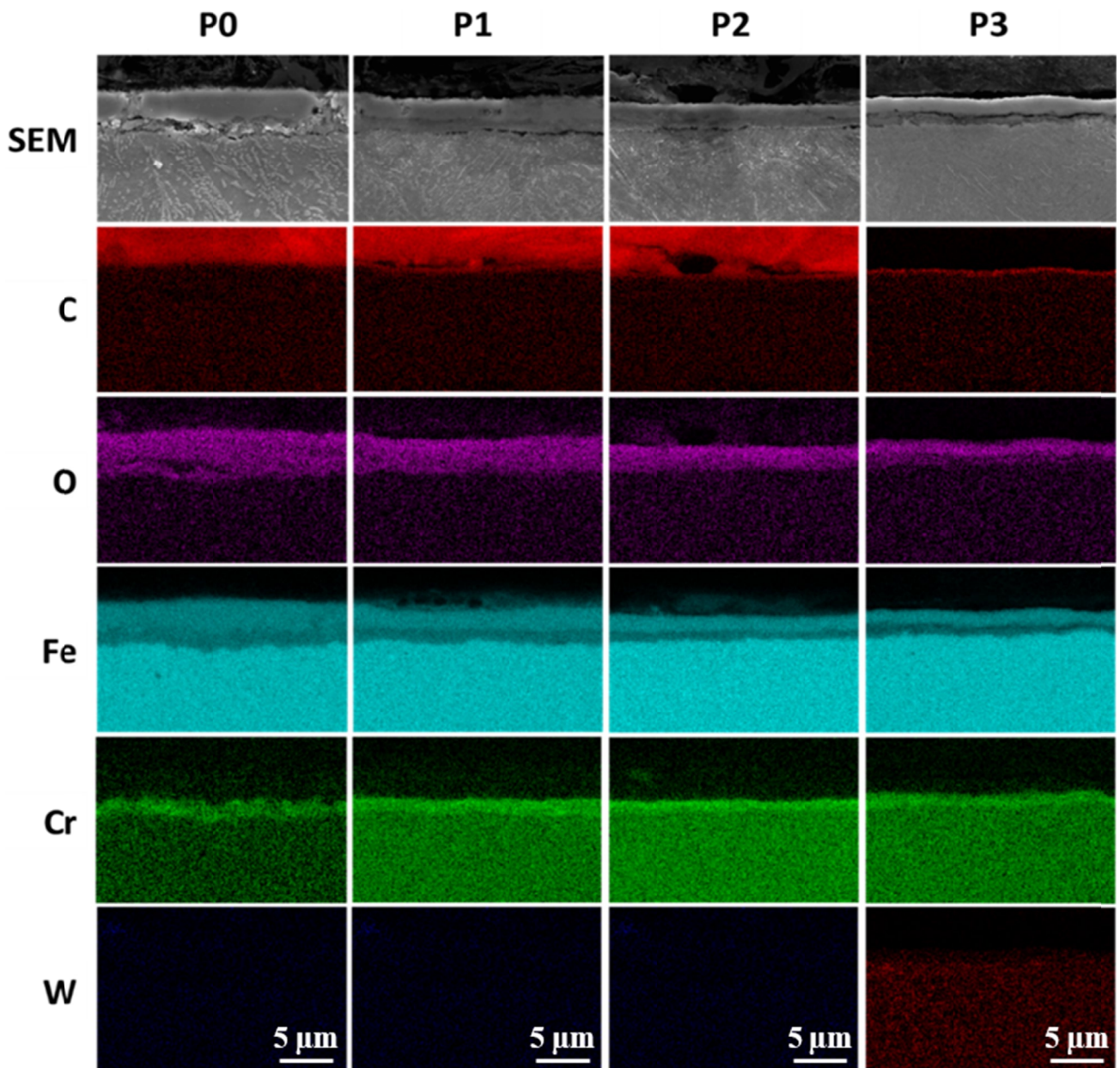


Fig. 4. Chemical compositions analysis of oxide layer of AISI 4140 (P0) and AISI 4140 Mod.s (P1, P2, P3) by EDS

At high temperatures, the oxide layer acts as a solid electrolyte, causing Fe ions to diffuse out of the oxide layer and oxidize to form an outer iron oxide layer. As the Cr content increases, the diffusion of Fe ions is slowed down, leading to a decrease in the rate of oxide layer formation [12,13].

W, like Cr, has the effect of delaying Fe ion diffusion due to the formation of the Cr_2WO_6 oxide layer and the doping of W in the Cr oxide layer [7]. Among the AISI 4140 Mod.s materials, P3, a material with 3Cr-1W, showed the highest oxidation resistance.

4. Conclusions

In this study, AISI 4140 Mod.s were manufactured with improved high-temperature strength, oxidation resistance, and wear resistance for application in automobile engine pistons. The microstructure and physical properties were analyzed based on the alloy compositions. Grain refinement, martensite formation, and stable carbide precipitation at high temperatures were designed through the addition of Mo, V, Nb, and Ni elements to increase high-temperature strength. These effects were confirmed through microstructure and tensile strength analysis. To enhance high-temperature oxidation resistance, elements such as Cr and W, which form a protective oxide layer, were added. The high-temperature oxidation resistance test demonstrated that the growth of the oxide layer was delayed as the contents of Cr and W increased. Compared to conventional AISI 4140, AISI 4140 Mod. exhibited improved high-temperature strength and high-temperature oxidation resistance. Following the evaluation of high-temperature wear resistance, AISI 4140 Mod. with enhanced strength, oxidation resistance, and wear resistance at high temperatures can be finally selected for automotive engine material.

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Conflicts of Interest

The authors declare no conflict of interest

Author Contributions

Hyo-Seong Kim: Investigation, Methodology, Writing – original draft & review & editing, preparation. **Moonseok Kang:** Investigation, Methodology, Writing – original draft, preparation. **Minha Park:** Investigation, Methodology. **Byung Jun Kim:** Investigation, Resources. **Byoungkoo Kim:** Conceptualization, Investigation, Funding acquisition, Supervision, Writing – original draft, preparation. **Yong-Sik Ahn:** Conceptualization, Supervision, Writing – review & editing.

REFERENCES

- [1] A.H. Meysami, R. Ghasemzadeh, S.H. Seyedein, M.R. Aboutalebi, *Mater. Des.* **31** (3), 1570-1575 (2010).
- [2] Y. Totik, R. Sadeler, H. Altun, M. Gavgali, *Mater. Des.* **24** (1), 25-30 (2003).
- [3] C.-Y. Jeong, *Mater. Trans.* **53** (1), 234-239 (2012).
- [4] G. Krauss, *Principles of heat treatment of steel*, Ohio 1980.
- [5] H. Bhadeshia, R. Honeycombe, *Steels: Picrostructure and Properties*, Oxford 2017.
- [6] B. Hwang, D.-W. Suh, *Korean J. Mater. Res.* **23** (10), 555-561 (2013).
- [7] D.G. Liu, L. Zheng, L.M. Luo, X. Zan, J.-P. Song, Q. Xu, X.Y. Zhu, Y.C. Wu, *J. Alloys Compd.* **765**, 299-312 (2018).
- [8] H. Tawancy, *Oxid. Met.* **45** (3), 323-348 (1996).
- [9] N. Birks, G.H. Meier, F.S. Pettit, *Introduction to the High Temperature Oxidation of Metals*, Cambridge 2006.
- [10] W.D. Klopp, *J. Less-Common Met.* **42** (3), 261-278 (1975).
- [11] N.J.P. Su-Yeon Cho, Chang-woong Je, *Engineer Metal*, Seoul 2013.
- [12] D. Jones, *Principles and Prevention of Corrosion*, New York 1992.
- [13] J.P. Lee, J.H. Hong, D.K. Park, I.S. Ahn, *J. Korean Powder Metall. Inst.* **22** (1), 52-59 (2015).