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Study on microstructure and oxidation behavior of Fe–xMn–14Al–8Ni–C alloy prepared by vacuum arc melting

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Abstract

In this study, Fe–xMn–14Al–8Ni–C alloy (x = 10, 15, 20, 25 wt.%) was prepared by vacuum arc melting method. The microstructure of this series of alloys and the oxidation behavior at 600 °C were studied. The conclusions are as follows: Fe–xMn–14Al–8Ni–C alloy mainly contains austenite phase, NiAl intermetallic compound phase and k-carbide phase. As the content of Mn increases, the amount of austenite increases while the amount of NiAl compound decreases. At the same time, the content of k-carbide phase precipitated at the interface between austenite and NiAl compound and inside austenite increases. The oxidation resistance results show that as the Mn content increases, the oxidation resistance of the alloy is improved. After oxidation, due to the difference in thermal stress and thermal expansion coefficient, the oxide film is mainly divided into two layers (when the Mn content is 10% and 15%, respectively), the outer oxides are Fe2O3 and a small amount of Mn2O3, and the inner oxides are mainly the mixture of Al2O3, Mn2O3 and NiO. When the Mn content increases to 20%, the oxide film is a three-layer, and a uniform dense oxide film mainly composed of Al2O3 appears at the junction with the substrate, which better prevents the further diffusion of oxygen in the air to the inside and protects the substrate. The surface oxide film is dense and stable, so it can prevent further oxidation. Therefore, the alloy with 25% Mn content exhibits the most excellent oxidation resistance.

Introduction

The combustion process of high power density diesel engine directly affects the power, emissions and economic performance [1, 2]. Among them, the movement of air flow in the combustion chamber and the mixing of oil and gas are the key factors affecting the combustion process of high power density diesel engines [2, 3]. With the further increase of power density, the working temperature and pressure of combustion chamber increase. As the most complex structure and load in the high power density diesel engine, the cylinder head is one of the most severe components in service conditions. High requirements for cylinder head material properties are put forward [4, 5]. Among them, excellent high-temperature oxidation resistance has become one of the important indicators to measure its superior performance. At present, alloy cast iron (higher density, about 7.3 g cm−3) is mostly used for cylinder heads of high-power density diesel engines. The bearing capacity above 500 °C is obviously reduced, especially the oxidation is intensified, which seriously reduces the fatigue performance of cast iron [4, 6].

Fe–Mn–Al alloys are not only low in density, but also high in strength, and also have excellent heat resistance. The alloys have broad research value in the automotive industry, military, and energy [7–9]. In 1958, Ham and Cairns took the lead in researching the low-cost Fe–34Mn–10A1–0.76C austenitic steel, and used this high-manganese steel to replace the more corrosion-resistant Cr–Ni stainless steel [10]. For corrosion resistance and high temperature oxidation resistance, the researchers use appropriate amounts of Al and Mn elements to replace Cr and Ni elements in stainless steel. Researchers from Hanba University studied Fe–Mn–Al–C alloys...
Fe–Alloy

The raw materials used in this research are industrial pure iron and found that in high-manganese steels, the alloys had low density (6.87 g cm\(^{-3}\)) and excellent plasticity and strength (strength–ductility balance is 60000% MPa)\(^{(11–13)}\). Mn element is an austenite phase forming element in Fe–Mn–Al alloy. The addition of Mn element can promote the formation of austenite phase, and it still exists in a large amount of austenite phase at room temperature. Adding an appropriate amount of Mn element to the Mn–Al alloy can suppress the formation of martensite phase during the phase transformation, and at the same time improve the alloy’s stacking fault energy, causing it to produce a large number of twins during deformation, which not only enhances the strength of the alloy, and to some extent improve the plasticity of the alloy\(^{(14)}\). It is generally known that the content of Mn in high manganese steel is higher than 12 wt.%. The atomic radius of Mn and Fe are similar, so there is no strong lattice distortion in the solid solution of Mn into austenite phase. The solid solution strengthening effect of Mn is not obvious compared with that of C element. At the same time, the structure will appear in the alloy, which will reduce its welding performance and thermal conductivity\(^{(15)}\). Researchers have studied the effect of Mn content on the mechanical properties of Fe–10Al–1C–Mn (solid symbols) and found that with increasing Mn content from 20 to 29%, Fe–10Al–1C–Mn (solid symbols) austenitic alloys showed virtually no change in the mechanical properties. This implies that Mn has little influence on the driving force of k-carbide formation in Fe–Al–Mn–C austenitic alloys\(^{(16, 17)}\). S H Kim et al developed a lighter, stronger and more flexible steel\(^{(18)}\). The key to this technology was the formation of new structural phases and compound phases during heat treatment process. Ni element was also added in their studies besides traditional elements such as Fe, C, Al and Mn. They found that during heat treatment Ni element will react with Al element to generate nano-sized NiAl phase with B2 structure; in addition, Al element addition to iron–based alloy can not only reduce the density of the alloy but also make the alloy have a certain oxidation resistance\(^{(18)}\).

Therefore, in this research, Fe–xMn–14Al–8Ni–C with different Mn contents (10, 15, 20, 25 wt.%) are prepared by vacuum arc melting. The microstructure and oxidation behavior of the series alloys have been systematically studied to provide theoretical direction for the development of new high-oxidation and low-density cylinder head materials.

**Experimental procedures**

The raw materials used in this research are industrial pure iron (purity \(\geq 99.99\)), electrolytic manganese flakes (purity \(\geq 99.99\)), high-purity aluminum particles (purity \(\geq 99.99\)), high-purity nickel particles (purity \(\geq 99.99\)) and Fe-5C alloy (purity \(\geq 99.99\)). The composition ratio of raw materials is shown in table 1. The non-consumable vacuum arc melting equipment produced by Shenyang Jinyan New Material Preparation Technology Co, Ltd was used for melting. Before melting the alloy, pure Ti which was put in a copper crucible in the furnace was melted first, so that the residual oxygen in the furnace was fully exhausted, and then the iron-based alloy began to be melted in other copper crucibles. During the melting process, the arc gun current was gradually increased from the minimum to 350A. At the same time, the electromagnetic stirring switch was turned on and each ingot was repeatedly melted 3 times. No slag formed during fusion. After each test, clean the furnace and copper crucibles with alcohol cotton.

The microstructure and elemental distribution of the Fe–xMn–14Al–8Ni–C alloys were investigated using VEGA II XMU scanning electron microscope (SEM) on an instrument equipped with an energy dispersive spectroscopy (EDS), 1600 Electron Prob Micro Analyzer (EPMA) and JEOL JEM-2010 type transmission electron microscope (TEM). Firstly, the alloy was made into 0.5 mm thin sheet by wire cutting, and the sheet was punched on the gauze until the thickness was reduced to 30 \(\mu\)m. After cleaning with alcohol, the specimen was cut into a 93 mm disc. Then, the sample is further thinned by Gatan691 precision ion thinning instrument until there is a small hole in the center of the sample. The phase identification of the composite was conducted by XRD-6000 x-ray diffraction (XRD, Shimadzu, Japan in which the scanning speed was 4° min \(^{-1}\), the range was

| Alloy | Electrolytic manganese (variable-x) | Industrial purity iron | High purity aluminum particles | High purity nickel particles | Fe-5C alloy |
|-----------------|-----------------------------------|------------------------|-------------------------------|-----------------------------|-------------|
| Fe–xMn–14Al–8Ni–C (10 \(\leq x \leq 25\)) | 10 | 48 | 14 | 8 | 20 |
|                | 15 | 43 | 14 | 8 | 20 |
|                | 20 | 38 | 14 | 8 | 20 |
|                | 25 | 33 | 14 | 8 | 20 |
20°–90°, the step length was 0.02) using Cu K alpha radiation. The oxidation experiment used RJ2-15-6 box-type resistance furnace, and the size of the oxidation sample was 8 × 8 × 3 mm. First, heating the box-type resistance furnace to 600 °C and heat preservation. After the furnace temperature was stable, putting the Al2O3 ceramic crucible (main chemical composition: Al2O3; type: 5 ml; the opening diameter: Φ 27 mm; height: 22 mm) in the furnace for different time and took it out to weigh it. Until the weight of the crucible was stable, putting the oxidized sample into the crucible and kept it warm. After a certain period of time, weighed the sample and oxidation curve of the mass increase with time was obtained. The oxidation samples were tested by INVIA micro-confocal laser Raman spectroscopy (laser wavelength of 785 nm, scanning range of 100–3200 cm⁻¹), combined with XRD test results to determine the surface composition of the oxidized samples. The oxidized surface of the sample with more typical oxidized morphology was protected and then cut and polished longitudinally to prepare an oxidized straight-cut sample. The morphology of the subsurface layer was observed using SEM. The oxidation mechanism was analyzed in conjunction with the SEM results of the oxidized surface.

Results and discussions

Microstructure analysis
After vacuum arc melting, waiting for 20 min and taking out the pie-shaped alloy ingot. The Fe–xMn–14Al–8Ni–C alloy ingot is shown in figure 1.

XRD analysis was carried out on Fe–xMn–14Al–8Ni–C alloy and the results are shown in figure 2. It can be seen from figure 2 that the alloy mainly contains austenite (JCPDS: 17-0333), NiAl intermetallic compound phase (JCPDS: 20-0019), and k-carbide phase (JCPDS: 48-1831). Among them, the austenite phase diffraction peaks at 42.1°, 48.9°, 71.8°, and 86.7°, and with the increase of Mn content, the intensity of the diffraction peaks at 42.1° and 86.7° gradually increased, and the austenite phase gradually increased; at 44°, 63.6°, 71.8° and 80.9° are NiAl phase diffraction peaks. When Mn content is 25%, the intensity of the diffraction peaks of the alloy at
63.6°, 71.8° and 80.9° gradually weakens, indicating that as the Mn content increases, the content of NiAl phase is reduced.

Figure 3 shows the microstructure of Fe–xMn–14Al–8Ni–C alloy, and table 2 is the corresponding point EDS results marked in figure 3. From figure 3(a) and its corresponding enlarged figure 3(b), it can be seen that there are continuously distributed network phases, and particles and strip phases of different shapes are distributed near the network phase. Combining the results of the energy spectrum analysis in table 2, when the content of Mn is 15%, the content of Mn and C in spectrum 1 is relatively higher than that in spectrum 3, and the content of Al and Ni is relatively lower than that in spectrum 3. While from spectrum 1 and spectrum 2, the element content is similar. It is inferred that the corresponding phase in point 1 is network austenite phase, point 2 is free austenite phase, and point 3 is NiAl phase. Previous studies have found that the enthalpy of formation of element content is similar. It is inferred that the corresponding phase in point 1 is network austenite phase. It is inferred that the corresponding phase in point 1 is network austenite phase, point 2 is free austenite phase, and point 3 is NiAl phase. Previous studies have found that the enthalpy of formation of NiAl phase is \( \Delta H = -22 \text{ KJ mol}^{-1} \), and the mixing enthalpy of Ni and Al atoms in this alloy system is the very small, so Ni atoms and Al atoms are the easily combined into NiAl compound [19, 20]. It can also be seen from figure 3(a) and its corresponding high magnification figure 3(b) that there are some elongated phases in the austenite phase, which is presumed to be k-carbide phase. At the same time, in figure 3(e) and the corresponding high magnification figure 3(f), combined with the spectrum 8 in table 2, it can be seen that the content of C element is higher at the white phase of the interface between austenite and NiAl compound, and the content of Mn and Al element is lower than that of the neighboring interface, the austenite phase is higher than the neighboring NiAl phase, presumably it is the K-carbides precipitated on the interface. Comparing figures 3(a), (c), (e) and (g), it can be seen that with the increase of Mn content, the content of network austenite phase gradually increases, and the content of NiAl phase gradually decreases. At the same time, the content of white phase at the interface of austenite and NiAl compound phase also gradually increases, mainly because the Mn element is an austenite forming element. In addition, as the Mn content increases, the k-carbide precipitated in the austenite phase also gradually increases. When the Mn content is 10%, the k-carbide less distributes in the austenite, while when the Mn content is 15%, the k-carbide content increases significantly, as the Mn content increase to 25%, more k-carbide precipitated from austenite. At the same time, k-carbide at the interface of austenite and NiAl compound also increases with Mn content increasing. It is because with the increase of Mn content, more contents of Mn element are dissolved into austenite during high temperature melting process. When the temperature is lowered, the solid solubility of austenite decreases and more Mn elements are precipitated from austenite and react with Fe, Al, C and other elements to generate k-carbide.

In order to further analyze the distribution of each element, Fe–25Mn–14Al–8Ni–C alloy is analyzed by EPMA, and the results are shown in figure 4. It can be seen from figure 4 that Mn and C elements are mainly distributed in the network phase area (austenite phase). For the phase adjacent to austenite, the content of Ni and Al elements is relatively high, combined with XRD and SEM test results, it can be concluded that the phase is NiAl compound. At the same time, the appearance of uneven distribution of Mn and Al elements is mainly due to the segregation of the alloy liquid during solidification.

The phase in Fe–25Mn–14Al–8Ni–C alloy is analyzed by TEM, and the results are shown in figure 5. Austenite phase, NiAl phase and k-carbide phase are contained in figure 5(a). k-carbide phase distributes at the interface between the austenite and the NiAl phase, it also can be seen that the interface between carbides and adjacent two phases is obvious and well combined. The diffraction spots corresponding to k-carbides is shown in figure 5(b), the matrix austenite phase and the interphase k-carbide phase can be seen in figure 5(c). Figure 5(d) is its corresponding diffraction spots. It can be seen that it consists of two sets of bright and dark spots, indicating that two different phases are distributed in the area. The analysis results show that the brighter set of spots corresponds to the austenite phase, while the set of slightly weaker spots corresponds to the k-carbide phase.

**Analysis of oxidation behavior**

Figure 6 is oxidation curve of Fe–xMn–14Al–8Ni–C alloy after holding at 600 °C for 125 h. It can be seen from figure 6 that the weight gain per unit area of the alloy with 10% Mn content changes most significant with oxidation time. As Mn content increases, the oxidation weight gain of the alloy gradually decreases. The final oxidation weight gain of alloys with Mn content of 10%, 15%, 20% and 25% are 0.52 mg cm\(^{-2}\), 0.44 mg cm\(^{-2}\), 0.35 mg cm\(^{-2}\) and 0.29 mg cm\(^{-2}\), respectively. Especially for the alloy with 25% Mn content, the final oxidation weight gain decreases by 79.31% compared with the alloy with Mn content of 10%.

X-ray diffraction test and Raman spectroscopy test analysis are performed on the surface of the specimen after oxidation, and the results are shown in figures 7(a) and (b), respectively. From the XRD test results of figure 7(a), it can be seen that the oxide layer of Fe–xMn–14Al–8Ni–C alloy after oxidation at 600 °C mainly contains Mn2O3 (JCPDS: 12-0141), Fe3O4 (JCPDS: 39-0238), and Al2O3 phases (JCPDS: 04-0877). Comparing figure 7(a) XRD patterns of the oxidation surface of Fe–xMn–14Al–8Ni–C alloy with different Mn contents, it
can be seen that with the increase of Mn content, the diffraction peaks of Mn$_2$O$_3$, Fe$_2$O$_3$, Al$_2$O$_3$ gradually strengthen, and the oxide content on the oxidation surface increased. To some extent, the oxide film can further prevent the diffusion of O atoms in the alloy, reducing the oxidation rate of the alloy. At the same time, the diffraction peak of the NiAl phase (JCPDS: 20-0019) appears, indicating that the NiAl phase still exists in the alloy.

Figure 3. SEM photograph of Fe–xMn–14Al–8Ni–C alloy. (a) 10%Mn; (b)10%Mn; (c)15%Mn; (d) 15%Mn. (e) 20%Mn; (f) 20%Mn; (g) 25%Mn; (h) 25%Mn.
sample after oxidation. From figure 7(b), the spectral peaks at 409.3 cm$^{-1}$, 476.4 cm$^{-1}$, 696.0 cm$^{-1}$, and 1300.0 cm$^{-1}$ represent Al$_2$O$_3$, and the spectral peaks at 312.2 cm$^{-1}$ and 647.1 cm$^{-1}$ represent Fe$_2$O$_3$ [21, 22]. Comprehensive XRD and Raman spectroscopy analysis results show that the main component of the oxidized surface is a mixture of Fe$_2$O$_3$ and Al$_2$O$_3$.

Figure 8 shows the oxidation surface morphology of Fe–xMn–14Al–8Ni–C alloy. It can be seen from figure 8 that there are a lot of cell-shaped protrusions on the oxidation surface of the alloy, and many granular oxides are contained in the cell surface layer. As the Mn content increases, the number of particulate oxides gradually increases, while the cellular oxides on the surface of the alloy decrease and tend to be smooth, indicating that the increase of Mn content can improve the oxidation resistance of the alloy, which may be due to reaction with O and Mn element generating element manganese oxide inhibits the oxidation expansion. Combined with the EDS analysis of the oxidation sample surface, it can be seen that the contents of O element and Mn element are high in the spectrum diagram 1, which is inferred to be Mn$_2$O$_3$. In figure 8(c), the reticular austenite region is more oxidized than NiAl compounds. It is mainly because there are fewer Al element in austenite, more Al element in NiAl compounds, and Al element are oxidized to form Al$_2$O$_3$ oxide film, preventing further oxidation.

| Mn content | Point | Element (wt.%) |
|------------|-------|----------------|
|            |       | Fe  | Mn  | Al  | Ni  | C   |
| x = 10     | 1     | 64.83 | 10.77 | 9.86 | 5.48 | 9.06 |
|            | 2     | 67.07 | 10.44 | 10.37 | 4.49 | 7.63 |
|            | 3     | 62.77 | 7.59  | 14.21 | 10.14 | 5.28 |
| x = 15     | 4     | 61.81 | 16.88 | 10.99 | 4.77 | 5.36 |
|            | 5     | 52.26 | 12.23 | 17.00 | 15.23 | 3.28 |
| x = 20     | 6     | 55.23 | 22.32 | 11.30 | 5.13 | 6.01 |
|            | 7     | 51.41 | 14.96 | 16.39 | 13.44 | 3.80 |
|            | 8     | 49.20 | 17.80 | 13.81 | 8.69 | 10.50 |
Alloy oxidation sections are further analyzed. Figure 9 shows the cross-section micrographs of Fe–xMn–14Al–8Ni–C alloy, and its corresponding EDS point analysis was shown in table 3. It can be seen from figure 9 that two oxide layers exist in each alloy after oxidation, which is due to the existence of thermal stresses produced by different coefficients of thermal expansion between oxides [23, 24]. Comparing the oxidation cross-sections of Fe–xMn–14Al–8Ni–C alloys with different Mn contents, it can be seen that the alloy oxide layers with Mn contents of 10% and 15% are thick and poor bonding with the substrate, oxygen was infiltrated locally and the oxidation of matrix is serious. It can be seen from table 3 that the oxide in spectrum 1 is mainly a mixture of Fe₂O₃ and a little Mn₂O₃, and it also can be seen that the oxide of this layer is relatively loose, and there are some

Figure 5. TEM image of Fe–25Mn–14Al–8Ni–C alloy. (a) interface microstructure; (b) k-carbide phase diffraction spots (c) austenitic microstructure; (d) austenite phase diffraction spots.

Figure 6. Fe–xMn–14Al–8Ni–C alloy oxidation curve.
voids inside. This is mainly due to the combination of C atom and O atom to form CO₂ during high temperature oxidation, which leads to decarburization and formation of voids [25]. The oxides in spectrum 2 is a mixture of a large amount of Al₂O₃ and a little Mn₂O₃ and NiO, and this oxide layer is relatively dense. It also indicates that there are still many Fe elements existence from EDS results. At high temperature, more Fe element contacts with O atom and reacts to form Fe₂O₃. In addition, during the oxidation process, there is a difference in chemical potential concentration difference between the gas in the furnace/oxide film and the oxide film/substrate. A thin oxide film is formed in the early stage, and the oxidation is mainly controlled by diffusion in later stage. The metal atoms in the sample diffuse outward, and O atom on the surface of the metal diffuses into the metal. All
that makes Al atoms combine with O atoms to form dense Al₂O₃, at the same time, Fe oxides and a small amount of Mn oxides rapidly nucleate and grow up in the Al₂O₃ gap, which is located outside the Al₂O₃ layer [26–28]. Compared with figures 8(a)–(c), it is found that as the Mn content increases, the thickness of the oxide layer gradually becomes thinner, indicating that the increase in the Mn content inhibits the expansion of oxygen atoms into the material. When the content of Mn is low, Al₂O₃ oxide film is not compact. During the oxidation process, oxygen atoms diffuse into the sample through the gaps in the oxide layer, resulting in increased oxidation of the sample. When the Al content is high, a dense layer of Al₂O₃ oxide film formed during oxidation. It can hinder the diffusion of O atoms into the metal and prevent the oxidation occurrence. The oxidation cross section analysis of this sample is consistent with its corresponding oxidation curve and the change of its oxidation surface.

Further, the line scan analysis results of the oxidation cross section of Fe–20Mn–14Al–8Ni–C alloy is shown in figure 10. It can be seen from figure 10 that the microstructure mainly consists of oxide layer and matrix, and the oxide layer is divided into three layers containing an outer layer (light-grey), an intermediate layer (dark-gray), and an inner layer (white). From the results of line scan, there are more Fe and O elements in the light-grey outer layer of the oxide layer, which is mainly enriched in Fe₂O₃ oxides; while the content of Al and O elements in the dark-gray inner oxide film is relatively high, and the content of Ni element, Fe element and Mn element is relatively low, indicating that this area is mainly Al₂O₃ oxides, and also contains a little other oxides containing NiO, Fe₂O₃ and Mn₃O₅. The white inner layer is relatively complete, mainly composed of Al-based oxide and Ni-based oxide, and it is well combined with the matrix, and there is no tendency for the oxygen element to diffuse inside. It can be seen that there are more Fe, Mn, Ni elements, and less Al and O elements in the matrix. In

### Table 3. EDS point analysis of Fe–10Mn–14Al–8Ni–C alloy in figure 9(a).

| Point | O     | Fe     | Mn     | Al     | Ni     |
|-------|-------|--------|--------|--------|--------|
| 1     | 29.88 | 63.17  | 6.95   | —      | —      |
| 2     | 33.59 | 27.89  | 15.03  | 13.91  | 9.58   |

Figure 9. Fe–xMn–14Al–8Ni–C alloy cross-section micrographs. (a) 10%Mn; (b) 15%Mn; (c) 20%Mn; (d) 25%Mn.
In this alloy system, the formation enthalpy of Al₂O₃ is the lowest, which indicates that during the oxidation process, O element diffuses from the surface of the sample to the inside, Al element diffuses from the inside to the outside, and combines with the O element to form the Al₂O₃ rich area (inner white area), which further prevents the diffusion of O atom.

Figure 11 is the schematic diagram of the oxidation process of Fe–xMn–14Al–8Ni–C alloy. It can be seen from figure 11 that with the increase of Mn content, the oxide layer becomes much denser, effectively preventing the further diffusion of oxygen into the matrix. Mn element can improve the oxidation resistance of the alloy by improving the stability and compactness of the oxide film, preventing the oxide film from falling off, thus slowing down the further oxidation of the alloy and improving the oxidation resistance of the alloy. Therefore, when the Mn content is 25%, the junction with the matrix alloy is a dense Al₂O₃ layer, which better prevents the further diffusion of oxygen in the air to the inside, protects the matrix from oxidation, and the surface oxide film is denser and stable, so the alloy has the most excellent oxidation resistance.

Conclusions

In this study, Fe–xMn–14Al–8Ni–C alloys with 10, 15, 20 and 25 wt.% Mn content were prepared by vacuum arc melting. The microstructure and oxidation behavior at 600 °C of the alloys were studied. The results and conclusions are as follows:
(1) Fe-xMn–14Al–8Ni–C alloy mainly contains austenite phase, NiAl intermetallic compound and k-carbide phase. With the increase of Mn content, the content of austenite and k-carbide increases, while the content of NiAl compound decreases.

(2) The results of oxidation test show that the oxidation weight gain of the alloy decreases with the increase of Mn content. The oxidation weight gain of Fe–25Mn–14Al–8Ni–C is 79.31% lower than Fe–10Mn–14Al–8Ni–C alloy.

(3) When Mn content is 10% and 15%, the oxide film is mainly divided into two layers—the outer oxide is Fe₂O₃ and a small amount of Mn₂O₃, and the inner oxide is consisted with Al₂O₃, Mn₂O₃ and NiO oxides. When the content of Mn up to 20%, the oxide film increases to three layers. A uniform and dense Al₂O₃ oxide film is formed at the junction with the matrix, which leads to Fe–25Mn–14Al–8Ni–C alloy has excellent oxidation resistance.

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