Article

Effect of W on the Impact-Induced Energy Release Behavior of Al–Ni Energetic Structural Materials

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Abstract: Energetic structural materials (ESMs) are an important class of military materials due to their good structural and energy-releasing characteristics. To improve the damage effect of metal–metal ESMs with good mechanical properties, W was added to the 48Al–52Ni composites, and the effect of W on the impact-induced energy release behaviors was investigated. The results showed that the hot-press process and the addition of W did not change the microstructure and surface state of the constituent particles, leading to a stable onset temperature of the Al–Ni intermetallic reaction in (48Al–52Ni)_{100-x}W_x composites. Meanwhile, the decrease in the contact area between Al and Ni in the composites with increased W content resulted in the decrease in reaction heat. During the impact process, the intermetallic reaction of W caused by the Al–Ni intermetallic reaction, as well as the oxidation reaction of Al and Ni caused by the brittle fracture along the weak interface, caused the released energy of (48Al–52Ni)₄₀W₆₀ to reach 2.04 kJ/g.

Keywords: energetic structural materials; Al–Ni composite; W; impact-induced reaction; energy release characteristic

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1. Introduction

Energetic structural materials (ESMs), also called reactive materials or multi-functional structural energetic materials (MSEM), are a class of novel structural materials [1]. ESMs are inert under ambient conditions, but reactions between components or with the atmosphere during application cause them to release amounts of chemical energy [2–4], leading to additional collateral damage [5]. ESMs with high density, excellent mechanical properties, and energy release characteristics seem to be the best materials for reactive fragments, shaped charges, and reactive bullets [4,6].

According to the component, ESMs are distinguished as three types: metal-fluoropolymer (such as Al–PTFE, etc.), metal-oxide (such as Al–Fe₂O₃, Al–CuO, Al–MnO₂, Al–MoO₃, etc.), and metal–metal (such as Al–Ni, Al–Hf, Al–Nb, Al–Ta, W–Zr, etc.) [7–10]. Among them, metal–metal ESMs have the best mechanical properties and can satisfy the needs of load-carrying structures. The damage effects of metal–metal ESMs became the key for application and were extensively investigated.

The damage effects of ESMs are dependent on kinetic energy and additional chemical energy released from impact-induced reactions. The improvement of damage effects has two components. The first one is composition optimization. For example, PTFE may be introduced in Al–Ni composites to promote an Al–PTFE reaction with a high heat reaction (55.67 kJ/g [11]) during impact loading [12]. Other oxides, e.g., CuO or MoO₃, which can react with Al and release a large amount of energy, were also added into the Al–Ni composites [13]. The addition of a non-metal improves the energy release performance of metal–metal ESMs, but it also leads to the collapse of mechanical properties.

The second way to improve the damage effects of metal–metal ESMs is the addition of a high-density component, which can greatly improve the kinetic energy of ESMs. For example, introducing W, whose density is 19.35 g/cm^3 , causes the combustion heat per unit volume to reach $21,150 \text{ cal/cm}^3$ [14] in Al–Ni composites [15]. The released energy per unit mass of Al–Ni composites with W can reach from 0.93 kJ/g to 2.23 kJ/g at an impact velocity of 1176 m/s [15]. The reaction mechanism of Al–Ni composites induced by heating has been widely investigated in previous work. Rogachev et al. presented that solid Ni was dissolved in Al and subsequently generated Ni–Al intermetallics, accompanied by the formation of a large amount of melt [16]. The combustion temperature of Al–Ni composites reached 1638°C during the formation of intermetallics [17]. The presence of high temperatures and a liquid phase is conducive to the dissolving of W in Al and Ni. Shchukin et al. reported that when W was partially dissolved in Al–Ni–W composites, the shape of the W particles was changed during the formation of Ni–Al intermetallic and finally obtained the pseudobinary eutectic Ni–Al–W system [17]. W remained a pure metal after the Al–Ni intermetallic reaction, indicating that W cannot take part in an intermetallic reaction. Although the positive effect of W on metal–metal ESMs, especially Al–Ni composites, has been demonstrated, the impact-induced reaction mechanism is not clear due to the difficulty in the analysis of the reaction process.

In this work, a classic metal–metal ESMs 48Al–52Ni (wt.%) composite is proposed. The effect of W on the impact-induced energy release behavior of Al–Ni ESMs is revealed from the microstructure, thermal reaction behavior, mechanical properties, and reaction product.

2. Materials and Methods

Al (spherical, $5 \mu\text{m}$, purity: 99.9%), carbonyl Ni (spherical, $5 \mu\text{m}$, purity: 99.8%), and W (spherical, $3 \mu\text{m}$, purity: 99.5%) raw powders were mixed by ball milling. These raw powders were purchased from Changsha Tianjiu Metal Material Co. Ltd. The ball-to-powder mass ratio, rotate speed, and milling time was 10:1, 250 r/min , and 20 min, respectively. The mixtures were pressed in an argon atmosphere under a pressure of 600 MPa at 673 K for 2 h to plates with a size of $50 \text{ mm} \times 30 \text{ mm} \times 13 \text{ mm}$. Some cylindrical samples ($\Phi 10 \text{ mm} \times 10 \text{ mm}$) and dish-like samples with a thickness of 0.3 mm were machined for subsequent experiments.

X-ray diffraction (XRD, Smartlab 9KW) with Cu $K\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) and scanning electron microscopy (SEM, JSM-6490LV) equipped with energy-disperse spectroscopy (EDS) were used to investigate the microstructure of composites. The valence state of the components was determined by X-ray photoelectron spectroscopy (XPS, Escalab Xi+) with Al $K\alpha$ rays (1486.6 eV). To calibrate the spectrum, a binding energy of 284.8 eV for the C1s peak was selected. Thermal reaction behaviors were conducted by thermogravimetric and differential scanning calorimetry (TG–DSC) (STA449F3, Jupiter, NETZSCH). The samples (about 6–10 mg) were heated to 1273 K at a heating rate of 10 K/min in a high-purity argon atmosphere (40 mL/min). Quasi-static compressive tests were carried out using an Instron 3369 testing system at a strain rate of 10^{-3} s^{-1} .

Impact-induced reaction behaviors were measured by ballistic tests, which were shown in our previous work [18]. The released energy caused by the impact of $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ (wt.%, $x = 0, 25, 40, 50$, and 60) composites were calculated by the classic empirical equation [19].

$$\Delta E = \frac{\Delta P V}{\gamma - 1} \quad (1)$$

where ΔE is released energy, ΔP is the maximum value of overpressure (the tested pressure minus the atmospheric pressure) in the test container, V is the volume of the test container, γ is the ratio of C_p to C_v . In the present work, V is 27 L , and γ is 1.4 .

3. Results and Discussion

3.1. Microstructure of $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ Composites

Figures 1 and 2 present the backscattered electron (BSE) images and XRD patterns of hot-pressed $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ ($x = 25, 40, 50$, and 60) composites. As shown in Figure 1, all these samples are composed of a black matrix phase and gray and white dispersed phases, although the volume fraction of black matrix decreases as the content of W in the composites increases from 25 wt.% to 60 wt.%. According to EDS results, the black, gray, and white regions are the pure metals Al, Ni, and W, respectively. They agree with the results shown in Figure 2, where only the diffraction peaks corresponding to Al, Ni, and W can be observed.

Figure 1. BSE images of the hot-pressed $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites. (a) $x = 25$; (b) $x = 40$; (c) $x = 50$; (d) $x = 60$.

Figure 2. XRD patterns of the hot-pressed $(48\text{Al}-52\text{Ni})_{40}\text{W}_{60}$ composites.

The presence of Al, Ni, and W in hot-pressed $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites indicates that the intermetallic-forming reaction between constituent elements did not occur because

the hot-pressed temperature of this work was 673 K, which is much lower than the reaction onset temperature for micro-sized Al–Ni, Al–W, and Ni–W composites [20–22].

Previous works have revealed that the formation of Al–Ni intermetallic is a diffusion-controlled solid reaction and is mainly decided by the interface between Al and Ni [18]. The effect of W on the Al/Ni interface is investigated by the XPS spectrum of Al and Ni. As shown in Figure 3, the binding energy of Al and Ni on the surface state, including ionic and atomic states, are independent of the presence of W. This suggests that W has a slight effect on the surface states of Al and Ni.

Figure 3. XPS patterns of Al₂p_{3/2} and Ni₂p_{3/2} spectra of (48Al–52Ni)₄₀W₆₀ composites at room temperature. (a) Al₂p_{3/2}; (b) Ni₂p_{3/2}.

3.2. Thermal Reaction Behavior of (48Al–52Ni)_{100-x}W_x Composites

Figure 4 presents the DSC curves of (48Al–52Ni)_{100-x}W_x composites protected with Ar gas. As can be seen, while the reaction onset temperature is stable in the region of 760–790 K, the total heat of reaction sharply reduces from ~1300 J/g to ~600 J/g as *x* increases from 0 to 60. Notably, an endothermic reaction peak at approximately 1140 K disappears when W is added.

Figure 5 shows the microstructure of (48Al–52Ni)_{100-x}W_x composites annealed at 790 K for 20 min. Aided by the results of EDS, the Al₃Ni and Al₃Ni₂ phase can be found in the annealed 48Al–52Ni composites (Figure 5a), while the Al and Ni particles disappear. By contrast, the Al₃Ni phase is absent in the annealed composites with W, where many W particles and Al₄W compounds are detected.

On the one hand, the onset temperature of exothermic reaction is dependent on the reaction type and the surface state of the reactants. According to the previous work, the exothermic reaction that starts at 760–790 K in (48Al–52Ni)_{100-x}W_x composites is the intermetallic reaction between Al and Ni. Meanwhile, the surface state of Al and Ni particles is rarely dependent on the content of W in the (48Al–52Ni)_{100-x}W_x composites, as shown in Figure 3. Thus, the onset temperature of (48Al–52Ni)_{100-x}W_x composites is relatively stable, as shown in Figure 4.

On the other hand, the heat released from the exothermic reaction is dependent on the contact area between reactants. As shown in Figure 1, the contact area between Al and Ni particles in the composites decreases as W content increases. Thus, the heat released in (48Al–52Ni)_{100-x}W_x composites is clearly reduced in the composites with a higher W content, as shown in Figure 4.

Moreover, according to the previous work [18], the endothermic reaction at approximately 1140 K in the Al–Ni composite is regarded as the melting of Al₃Ni. The absence of the melting of Al₃Ni indicates the absence of the Al₃Ni phase and the change of Al–Ni reaction in the composites with W. The analysis for the reaction product of (48Al–52Ni)_{100-x}W_x composites (Figure 5) agrees with this phenomenon.

Figure 4. DSC curves of $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites (a) $x = 0$; (b) $x = 25$; (c) $x = 40$; (d) $x = 60$.

Figure 5. Microstructures of annealed $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites. (a) $x = 0$; (b) $x = 25$; (c) $x = 40$; (d) $x = 60$.

According to the report of the reaction mechanism for Al–Ni composites [23], the Al_3Ni phase first formed at the Al/Ni interface, and then the Al_3Ni_2 phase was generated through the diffusion of Ni. Two exothermic peaks for all $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites at 700–1000 K (Figure 4) and the presence of Al_3Ni_2 in all annealed composites jointly demonstrates the same Al–Ni intermetallic-forming reaction process in the composites with W.

Al reacts with Ni accompanied by distinct self-heating in an equiatomic mixture of Ni–Al powders, producing a large amount of liquid phase in the form of Ni–Al melt [21]. This self-heating phenomenon causes the temperature to reach 1550 reported in [17]. The temperature is high enough to dissolve W in Al and Ni phases, and the solubility of W is dependent on the diffusion of metals at W/Al and W/Ni solid–liquid interface. When the temperature is above 1300 °C, the solubility of W in the Al phase increases to 15 At.% [24,25], while when the temperature rises to 1510 °C, the solubility of W in the Ni phase increases to 17.5 at.% [24,25]. In other words, as long as the chemical reaction between Al and Ni initiates, the Al and Ni phases can dissolve W. Some Al–W and Ni–W compounds generate with sufficient diffusion time [26]. Thus, the presence of Al_4W and Ni_4W , as shown in Figure 5, is the result of diffusion and reaction for Al–W and Ni–W systems. The reaction of Al–W would consume a certain amount of Al, which changes the ratio between Al and Ni, resulting in the absence of Al_3Ni , as shown in Figures 4 and 5.

3.3. Impact-Induced Energy Release Behaviors of $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ Composites

Figure 6 shows the quasi-static overpressure (the tested pressure minus the atmospheric pressure) in the test chamber caused by the impact of $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ ($x = 0, 25, 40, 50$, and 60) composites with a velocity of 1200 m/s. As can be seen from Figure 6a,c, the penetration of the $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites causes an obvious flame and a sharp increase in gas pressure in the test chamber within 10 ms. Subsequently, the gas pressure in the test chamber gradually reduces to atmospheric pressure due to the presence of the bullet hole connecting with the environment (Figure 6b) and the temperature rise caused by the reaction of the $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites vanishes. The dependence of released energy calculated by Equation (1) on the W content in $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites are shown in Figure 6d. The released energy caused by the penetration of $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites increases from 0.83 kJ/g to 2.04 kJ/g as x increases from 0 to 60.

Figure 7 presents the XRD pattern and SEM images of the collected fragments of the $(48\text{Al}-52\text{Ni})_{40}\text{W}_{60}$ composites. In the first XRD pattern (Figure 7a), a set of strong diffraction peaks are identified as W, while other weak diffraction peaks can be attributed to Al_3Ni_2 , Ni_4W , Al_2O_3 , NiWO_4 , and oxide-scale of W, respectively, but not Al and Ni. The collected fragments are divided into two types: irregular and spherical fragments. According to EDS results shown in Table 1 and Figure 8, it can be known that the irregular fragments are composites of Al_3Ni_2 (dark gray region), W (white spherical region), and Al_4W (gray region), and the spherical fragments are composites of W (white spherical region), Ni_4W (gray region), and Al_2O_3 (black spherical region).

Table 1. EDS results of Figure 7 (at.%).

	Al	Ni	W	O	Phase
A	-	6.0	94.0	-	W
B	64.7	35.3	-	-	Al_3Ni_2
C	78.9	-	21.1	-	Al_4W
D	57.0	-	-	43.0	Al_2O_3
E	-	79.3	20.7	-	Ni_4W

Figure 6. Energy release behaviors under high-speed impact loading. (a) The flame captured by a high-speed camera; (b) bullet hole; (c) quasi-static pressure inside test chamber; (d) relationship between released energy and the W content.

Figure 7. XRD patterns and SEM images of recovered $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ fragments. (a) XRD patterns; (b) irregular fragments; (c) magnified image for (b); (d) spherical particle; (e) magnified image for (d).

Combined with the reaction products generated in Ar (Figure 5), it can be known that Al and Ni in $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ composites react completely during impact loading, including the intermetallic reactions and oxidation reaction. The energy released by the reaction of Al and Ni makes some W participate in an intermetallic reaction. Compared with Al and Ni, W has higher latent heat but lower activity. Thus, the reaction of W exactly enhances the released energy of the Al–Ni composites with W.

Figure 8. Elemental mapping of recovered $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ fragments.

On the other hand, the mechanical properties and fracture mechanism also have a significant influence on the released energy of composites. As shown in Figure 9, which shows the crack propagation for the compared Al–Ni composites with or without W, the deformation in the 48Al–52Ni composites mainly occurs in the Al matrix. The mechanical properties of the composites are thus decided by those of the Al matrix. When W is introduced, the deformation and fracture in the $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ composites are concentrated at the interface between Al and W. The mechanical properties of the $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ composites are thus decided by the binding between Al and W. As shown in Figures 1 and 2, the binding between Al and W in the $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ composites is a weak molecular binding without the formation of ionic bond or covalent bond. Therefore, the mechanical properties, especially the fracture strain, are clearly reduced in the composite with W, as shown in Table 2. The brittle fracture means more fresh surface for the oxidation of constituent metals and a higher amount of energy released by the oxidation reaction. Therefore, the intermetallic reaction of W and the higher oxidation reaction of Al co-contribute to the higher amount of released energy of the composites with W, as shown in Figure 6d.

Figure 9. Crack propagation for $(48\text{Al}–52\text{Ni})_{100-x}\text{W}_x$ composites during compression. (a) $x = 0$; (b) $x = 60$.

Table 2. Mechanical properties of $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites.

x	$\rho/\text{g}\cdot\text{cm}^{-3}$	TMD/%	σ_c/MPa	σ_{cf}/MPa	$\varepsilon_{cf}/\%$
0	4.18	98.8	289 ± 16	480 ± 31	3.8
25	4.13	98.20	296 ± 18	391 ± 14	1.8
40	5.90	96.58	297 ± 10	381 ± 8	1.7
50	6.66	96.50	323 ± 6	396 ± 6	1.7
60	7.44	94.89	318 ± 5	400 ± 2	1.7

σ_c is compressive yield strength; σ_{cf} is compressive fracture strength; ε_{cf} is compressive fracture strain.

4. Conclusions

In this work, $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites without intermetallics were prepared successfully by ball-milling and hot-pressing. The effects of W on the microstructure, thermal reaction behaviors, and impact-induced energy release behaviors of Al–Ni ESMs were systematically investigated. The following conclusions were obtained:

- The introduced spherical-like W particles were randomly distributed in the continuous Al matrix. The interfaces between Al and Ni decreased, while those between Al and W increased as W content in the composites increased. The surface state of Al and Ni were independent of the addition of W.
- The stable surface state of Al and Ni led to similar onset temperatures of the Al–Ni intermetallic reaction in $(48\text{Al}-52\text{Ni})_{100-x}\text{W}_x$ composites. The decrease in contact area between Al and Ni resulted in the decrease in reaction heat.
- It was shown that the energy released by the Al–Ni intermetallic reaction could lead to the W–Al intermetallic reaction and result in the absence of Ni_3Al during the annealing at Ar atmosphere.
- During the impact process, W–Al and W–Ni intermetallic reactions could be caused by Al–Ni intermetallic reactions as well as the oxidation reaction of Al and Ni. Meanwhile, more Al and Ni were oxidized due to more fresh metal surface related to the brittle fracture along the weak interface between Al and W particles. The participation of W–Al and W–Ni in the intermetallic reaction, as well as the higher oxidation reaction of Al and Ni, co-contributed to the greater amount of released energy of $(48\text{Al}-52\text{Ni})_{40}\text{W}_{60}$.

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