

Effect of Vanadium Addition on the Microstructure and Mechanical Properties of Si–Mo Ductile Iron at Room and Elevated Temperatures

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Effect of Vanadium Addition on the Microstructure and Mechanical Properties of Si–Mo Ductile Iron at Room and Elevated Temperatures

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Abstract: The effects of V addition on the microstructure and mechanical properties of Si–Mo ductile iron at room temperature and at elevated temperatures were investigated. V addition reduced the pearlite fraction and altered the carbide morphology in the as-cast microstructure, transforming fishbone-like $(\text{Mo,Fe,Si})_6\text{C}$ into dispersed, blocky $(\text{V,Mo})\text{C}$. After exposure at 800 °C, both $(\text{V,Mo})\text{C}$ and $(\text{Mo,Fe,Si})_6\text{C}$ carbides showed coarsening accompanied by blunting and partial spheroidization. With increasing V content, the oxide scale formed at 800 °C became thicker, less dense, and more susceptible to internal oxidation. The room-temperature strength increased initially and then decreased with increasing V content, reaching a maximum of 651 MPa at 0.37% V, approximately 8% higher than that of the V-free specimen. In contrast, the high-temperature strength at 800 °C increased monotonically with V content, reaching 59.7 MPa at 0.56% V, approximately 23% higher than that of the V-free specimen. The creep-rupture life at 800 °C also increased with V addition, extending from 0.9 h in the V-free specimen to 9.7 h at 0.56% V (approximately a 10-fold improvement). The pre-oxidation fatigue life at 800 °C increased initially and then decreased with increasing V content, peaking at 1.278×10^6 cycles at 0.37% V, approximately 1.3 times that of the V-free specimen.

Keywords: Si–Mo ductile iron; microstructure; V alloying; mechanical properties; $(\text{V,Mo})\text{C}$ carbide

Introduction

Exhaust-gas temperatures in commercial vehicle engines are steadily approaching 800 °C and higher, driven by advanced powertrain technologies such as high-peak-pressure diesel engines, natural-gas engines, and hydrogen/ammonia engines ^[1,2]. This trend places increasingly stringent requirements on exhaust-manifold materials. In service, damage from high-temperature fatigue, creep, and oxidation accumulates continuously in the manifold, and failure occurs when the accumulated damage exceeds the material's mechanical capacity. Conventional medium-Si–Mo ductile iron (3.5%–4.5% Si, 0.5%–1.5% Mo) is therefore no longer sufficient for future engine applications. Alloying has consequently become a key strategy for enhancing the mechanical properties, creep resistance, fatigue performance, and oxidation resistance of Si–Mo ductile iron.

Lekakh ^[3] and Ekström ^[4] reported that Cr improves the oxidation resistance of Si–Mo ductile iron. However, Cr addition increases the pearlite fraction ^[5], thereby reducing ductility. Moreover, pearlite decomposition at elevated temperatures can generate substantial thermal stresses in components. Celik ^[6,7] partially replaced Mo with Nb and added 1% Nb and 0–4% Al to ductile iron containing 4% Si, obtaining improved oxidation resistance relative to conventional Si–Mo ductile iron. However, de Magalhaes ^[8] reported that Nb addition did not improve either the room-temperature or high-temperature strength of Si–Mo ductile iron, while reducing room-temperature elongation by 30%. The Swiss company GF developed an Al-containing SiMo1000 grade, which demonstrates markedly improved high-temperature performance and has been widely applied in heat-resistant components such as automotive exhaust manifolds ^[9,10]. Ibrahim ^[11] added 3% Al to low-Si SiMo ductile iron and observed a moderate strength increase; however, nodularity, elongation, and impact toughness deteriorated significantly. Similarly, Lekakh ^[12] revealed that Al addition reduced graphite nodularity and ductility, which in turn promoted crack initiation and propagation during thermal cycling of Si–Mo ductile iron. Ekström ^[13] reported that Ni increased the low-cycle fatigue life of Si–Mo ductile iron at 250 °C but reduced elongation. Abdelrahim ^[14] used Thermo-Calc to estimate the eutectoid transformation temperature of alloyed Si–Mo ductile iron containing 0.63% Ni and found it to be approximately 50 °C lower than that of the unalloyed grade, indicating that even small Ni additions can reduce thermal stability. Ekström ^[4] further showed that adding 1% Ni to Si–Mo ductile iron led to severe oxidation at 800 °C because Ni disrupted the continuity of the protective SiO₂ film.

Vanadium (V) is a strong carbide-forming element with excellent thermal stability and is therefore expected to improve the high-temperature performance of Si–Mo ductile iron. Yamauchi et al. ^[15] increased the V and Si contents of conventional Si–Mo ductile iron. The resulting tensile strength at 800 °C increased by approximately 51% compared with that of the conventional grade, while the thermal-fatigue resistance exceeded that of the more expensive Ni-resist cast iron. However, this improvement was partly attributed to the increased Si content; therefore, the individual effect of V on the microstructure and properties remains unclear. Chao et al. ^[16] investigated the effects of Nb and V on the microstructure and properties of Si–Mo ductile iron and found that V promoted pearlite formation and induced the precipitation of abundant V-rich carbides in the matrix. These microstructural changes significantly enhanced the high-temperature mechanical properties, and the strengthening effect of V exceeded that of Nb. However, that study focused only on mechanical properties and did not comprehensively evaluate the high-temperature performance of Si–Mo ductile iron under service-relevant conditions.

Current research on V-containing cast iron has primarily focused on conventional ductile iron ^[17–20], whereas studies of its effects on the microstructure and properties of Si–Mo ductile iron remain limited. In particular, the microstructural evolution and strengthening mechanisms of V-containing Si–Mo ductile iron at 800 °C have rarely been reported. Therefore, the present work investigates the effects of V content on the room-temperature and high-temperature microstructures, oxidation behavior, creep behavior, and fatigue performance of Si–Mo ductile iron. The underlying mechanisms are also analyzed. This study is expected to provide a theoretical basis for developing heat-resistant exhaust-manifold materials for commercial-vehicle engines operating at 800 °C.

Methods

Specimen Preparation

The raw materials comprised high-purity pig iron, steel scrap, ferrosilicon, ferromolybdenum, and ferrovanadium. Melting was performed in a 500-kg medium-frequency induction furnace. The melt composition was adjusted, and the tapping temperature was controlled at 1520 ± 10 °C. A nodulizer (1.3% FeSiMg7RE3Ca2.5) and inoculant (0.3% 75FeSi) were placed at the bottom of the ladle, and the molten iron was poured into the ladle for nodulization and inoculation. The treated melt was cast into 25 mm Y-block molds (Figure 1) at a pouring temperature of 1390 ± 10 °C, with secondary-stream inoculation using ferrosilicon during pouring. After cooling, specimens were extracted from the bottom shadow region of the standard Y-block for microstructural characterization and mechanical testing. Four alloys with different V contents were prepared; their chemical compositions are listed in Table 1.

Table 1. Chemical compositions of V-alloyed Si–Mo ductile iron (wt%)

样品	C	Si	Mn	P	S	Mo	V	Mg
S1	3.20	4.16	0.20	0.038	0.009	0.91	0	0.038
S2	3.15	4.18	0.21	0.032	0.010	0.88	0.19	0.039
S3	3.21	4.15	0.20	0.033	0.009	0.88	0.37	0.042
S4	3.16	4.25	0.23	0.032	0.010	0.90	0.56	0.041

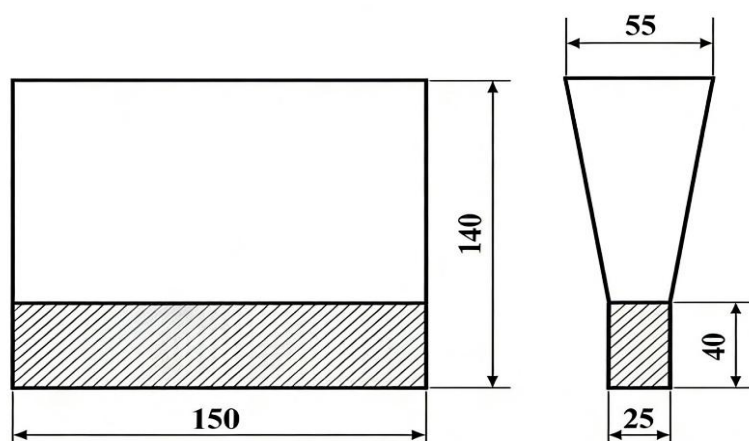


Figure 1. Dimensions of the standard Y-block specimen (mm).

Microstructural Characterization

Specimens were ground and polished and then etched with 4% HNO₃ in alcohol. The graphite morphology and matrix microstructures in Si–Mo ductile iron with different V contents were examined using an Axio Observer Z1M optical microscope. The carbide morphology and surface oxide film were characterized using a SU-70 field-emission scanning electron microscope (FE-SEM). Elemental compositions were analyzed by energy-dispersive spectroscopy (EDS; OXFORD).

Room-Temperature Mechanical Property Testing

Brinell hardness was measured using a 400HBS3000 digital hardness tester with a load of 7350 N, a 5-mm indenter, and a dwell time of 20 s. Room-temperature tensile testing was conducted on an ETM305D electronic universal testing machine at a strain rate of 0.0067 s^{-1} ; the specimen geometry is shown in Figure 2a. At least three specimens were tested for each alloy, and the average

value is reported. Tensile fracture surfaces were examined by FE-SEM.

High-Temperature Oxidation Test

Cylindrical specimens ($\phi 12.7 \text{ mm} \times 1.5 \text{ mm}$) were machined. Both end faces were ground, and the specimens were placed in a chamber electric resistance furnace. Oxidation testing was performed at 800 °C for 60 h, after which the specimens were furnace-cooled to room temperature. The specimens were sectioned longitudinally, mounted, ground, and polished, and the cross-sectional microstructure was subsequently analyzed.

Differential Scanning Calorimetry (DSC) Analysis

Differential scanning calorimetry (DSC) was performed using a NETZSCH STA449F3 synchronous thermal analyzer under a protective nitrogen atmosphere. Measurements were conducted from room temperature to 1200 °C at a heating rate of 5 °C/min. Approximately 20 mg of each specimen was placed in an aluminum crucible, and the heat-flow difference as a function of temperature was recorded under programmed temperature control. Thermal parameters—including the pearlite decomposition temperature, eutectoid transformation temperature, carbide dissolution temperature, and eutectic temperature—were determined from the DSC curves.

High-Temperature Tensile Strength and Creep-Rupture Tests

High-temperature tensile strength and creep-rupture properties at 800 °C were evaluated using a CMT5205 high-temperature tensile testing machine. Specimen dimensions are shown in Figure 2b. For the tensile tests, specimens were held at 800 °C for 20 min prior to loading. The strain rate was 0.0014 s^{-1} . At least three specimens were tested for each alloy, and the mean value was calculated. Fracture surfaces were subsequently examined by FE-SEM.

For the creep-rupture tests, the specimens were held at $800 \pm 2 \text{ °C}$ for 60 min prior to loading. A constant tensile stress of 25 MPa was applied.

Pre-Oxidation High-Temperature Fatigue Test

Fatigue specimens were machined to the dimensions shown in Figure 2c. Specimens were pre-oxidized in a box-type resistance furnace at 800 °C for 60 h to form an oxide film on the surface. High-temperature tension–compression fatigue tests were then performed at $700 \text{ °C} \pm 2 \text{ °C}$ using a QBG-250 high-frequency fatigue testing machine. A stress ratio of $R = -1$ was used. The stress amplitude was 50 MPa, and the test frequency was 80 Hz. The number of cycles to fracture was recorded for each specimen. At least five specimens were tested for each alloy, and the mean value was reported.

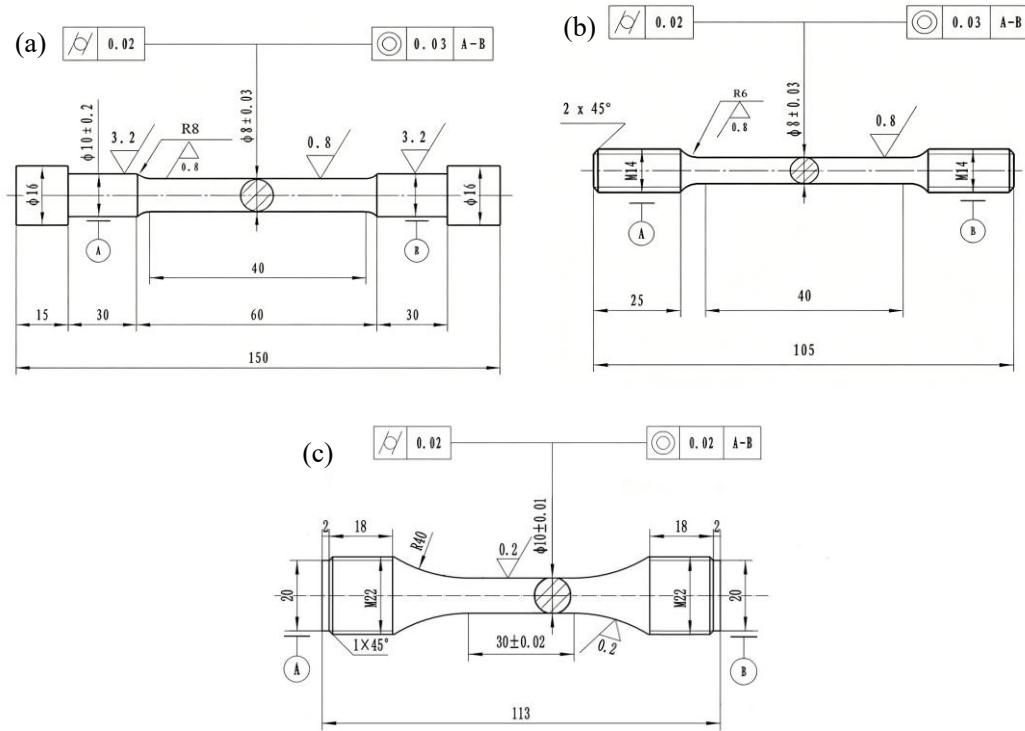
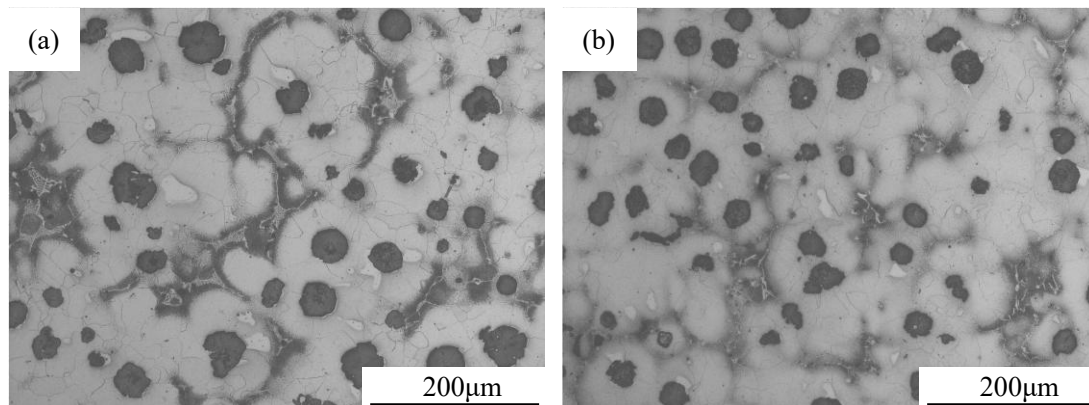


Figure 2. Dimensions of tensile specimens (mm): (a) room-temperature, (b) high-temperature, and (c) pre-oxidation high-temperature fatigue.

Results and Discussion

Microstructure

Figure 3 presents the graphite morphology and matrix microstructure of Si-Mo ductile iron with different V contents. As shown in Figure 3a-d, neither graphite nodularity nor graphite size changed appreciably with increasing V content. By contrast, the pearlite fraction in the matrix decreased markedly as the V content increased.



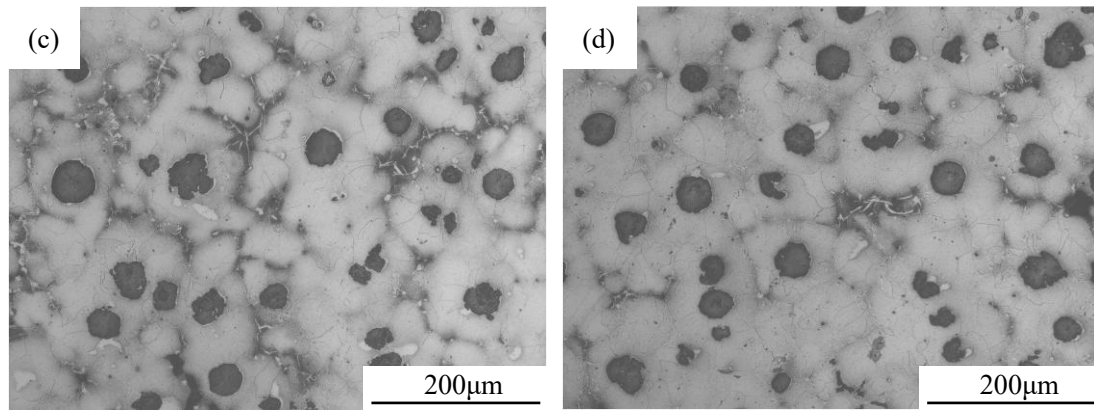


Figure 3. Microstructure of Si-Mo ductile iron with different V contents: (a) 0%, (b) 0.19%, (c) 0.37%, and (d) 0.56%.

V is a strong carbide-forming element rather than a surface-active element and therefore exerts little influence on the nodulizing effect of Mg in molten iron. Accordingly, graphite nucleation and spheroidal growth are not adversely affected by V addition. In contrast, pearlite formation during the eutectoid transformation depends on C diffusion and cementite precipitation. After eutectic solidification, V exhibits limited solid solubility in the austenite matrix. Upon cooling, the solubility of V decreases and fine carbides precipitate within austenite grains. This precipitation reduces the solubility of C in austenite, thereby decreasing C activity and diffusivity, suppressing eutectoid pearlite formation [22]. Consequently, the pearlite fraction decreases significantly with increasing V content.

Figure 4 shows the carbide morphology and EDS elemental mapping results of Si-Mo ductile iron with different V contents. In the V-free specimen, carbides predominantly formed a continuous fishbone-like morphology along grain boundaries, and EDS indicated Mo enrichment in these carbides. With increasing V content, the fishbone-like structure gradually diminished and carbides became progressively refined into a blocky morphology. Intragranular carbides increased in number and were more uniformly distributed. EDS mapping indicated that, in the specimen containing 0.19% V, both V and Mo were present in the blocky carbides, whereas the fishbone-like carbides contained only Mo. The fraction of blocky carbides increased with further increases in V content. In the 0.56% V specimen, the spatial distributions of V and Mo in the carbides were highly coincident.

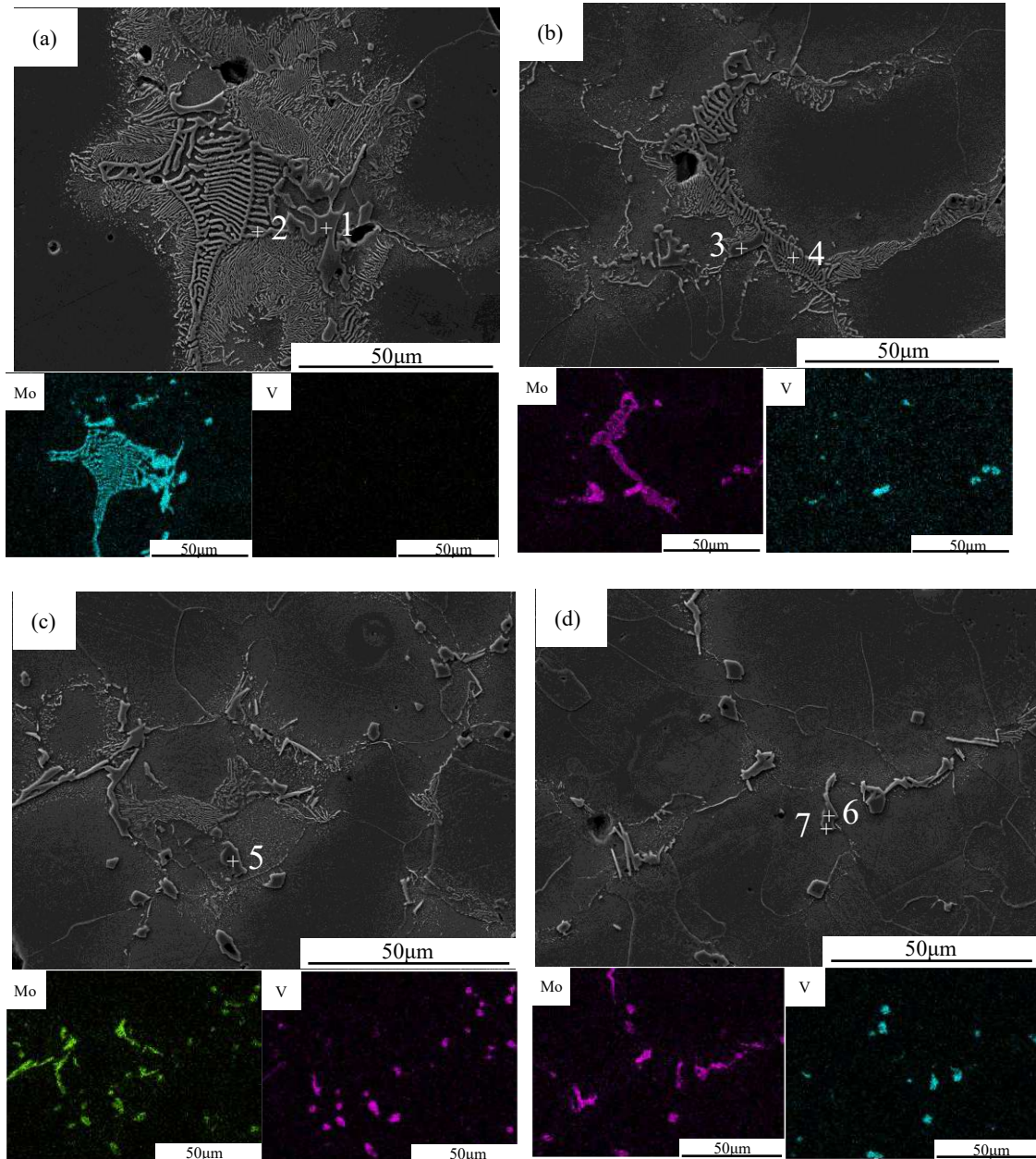


Figure 4. Carbide morphology and EDS analysis of Si-Mo ductile iron with different V contents: (a) 0%, (b) 0.19%, (c) 0.37%, and (d) 0.56%

During solidification of Si-Mo ductile iron, Mo segregates to the liquid phase. At the final stage of eutectic solidification, Mo-rich M_6C carbides precipitate with a fishbone-like or network morphology [23]. The addition of V modifies this precipitation behavior. Because V has a strong affinity for C, V-containing carbides can form at an earlier stage of solidification [24]. Owing to the similarity in crystal structure between Mo and V, Mo can dissolve into the V-carbide lattice, forming MC-type complex carbides [25–27]. These V-rich carbides exhibit greater thermodynamic stability and a higher precipitation temperature than M_6C [28,29]. Thus, during solidification, V combines with C in the liquid to generate numerous high-melting-point carbide nuclei, which also deplete Mo from the liquid. As a result, the continuous growth of Mo-rich M_6C carbides is suppressed, and the carbide morphology shifts from fishbone-like to discontinuous blocky forms.

To further clarify the phase constitution and elemental distribution of carbides in Si–Mo ductile iron with different V contents, EDS point analyses were performed in the representative regions marked in Figure 4, and the results are summarized in Table 2. In the V-free specimen, Point 1 was located at the center of a carbide and contained 28.25 at% Mo, 22.18 at% C, 37.59 at% Fe, and 11.21 at% Si. Point 2 was located at the edge of the same fishbone-like carbide and, relative to Point 1, showed a lower Mo content (19.62 at%) and a higher Fe content (50.38 at%), accompanied by slight decreases in C and Si. On the basis of the fishbone-like morphology, atomic ratios, and prior reports on alloy carbides, this continuous phase was identified as the eutectic carbide $(\text{Mo,Fe,Si})_6\text{C}$ [30,31]. Point 3 was located at the center of a blocky carbide in the 0.19% V specimen; the V and Mo contents were 31.77 at% and 21.85 at%, respectively, with 42.23 at% C and an extremely low Si content. Consistent with its blocky morphology, atomic ratios, and relevant literature, this carbide was identified as $(\text{V,Mo})\text{C}$ [32–34]. Point 4 was located within a fishbone-like carbide in the 0.19% V specimen; the C, Si, Mo, and Fe contents were comparable to those at Point 2, and V was negligible (1.02 at%). This region was therefore also identified as $(\text{Mo,Fe,Si})_6\text{C}$. Point 5 was positioned at the center of a blocky carbide in the 0.37% V specimen; its elemental ratios were similar to those at Point 3, indicating $(\text{V,Mo})\text{C}$. Points 6 and 7 were located at the center and edge, respectively, of a blocky carbide in the 0.56% V specimen, and both exhibited compositions consistent with $(\text{V,Mo})\text{C}$. Notably, the carbide center was enriched in V, whereas the edge showed reduced V and increased Mo, Fe, and Si contents.

Table 2. EDS analysis results of the points marked in Figure 4

Point	C(at%)	Si(at%)	Fe(at%)	Mo(at%)	V(at%)	Type of phase
1	22.18	11.21	37.59	28.25	-	$(\text{Mo,Fe,Si})_6\text{C}$
2	19.09	10.91	50.38	19.62	-	$(\text{Mo,Fe,Si})_6\text{C}$
3	42.23	0.23	3.85	21.85	31.77	$(\text{V,Mo})\text{C}$
4	20.44	10.22	51.30	16.80	1.02	$(\text{Mo,Fe,Si})_6\text{C}$
5	40.59	0.32	6.11	15.94	36.77	$(\text{V,Mo})\text{C}$
6	41.00	0.15	3.61	11.11	44.04	$(\text{V,Mo})\text{C}$
7	40.21	1.79	16.35	26.77	14.76	$(\text{V,Mo})\text{C}$

V exhibits a strong affinity for C and forms high-melting-point VC nuclei during the early stages of solidification. Because Mo and V are both Group VB elements with similar atomic radii, Mo can dissolve in the VC lattice, leading to the formation of complex $(\text{V,Mo})\text{C}$ carbides. As solidification proceeds, Mo becomes progressively enriched in the residual liquid, and carbides that form later therefore incorporate higher Mo contents. Consequently, a compositional gradient develops, with V decreasing and Mo increasing from the carbide core toward the periphery. With further solidification, Mo in the remaining liquid far exceeds V, rendering the liquid Mo-rich, and promoting the precipitation of Mo-rich carbides. Accordingly, the carbide population gradually transitions toward M_6C -dominated phases. In specimens with higher V contents, nucleation of $(\text{V,Mo})\text{C}$ is strongly dominant. Most of the Mo in the liquid is consumed during $(\text{V,Mo})\text{C}$ growth. As a result, the M_6C eutectic reaction is fully suppressed, and the carbides are predominantly blocky $(\text{V,Mo})\text{C}$.

Room-Temperature Mechanical Properties and Fracture Morphology

Figure 5 shows the room-temperature mechanical properties of Si–Mo ductile iron with different V contents. Increasing the V content from 0 to 0.56% produced an overall increase in room-temperature tensile strength, with a maximum average value of 651 MPa at 0.37% V, approximately 8% higher than that of the V-free specimen (604 MPa). A further increase to 0.56% V led to a slight reduction in strength to 649 MPa. In contrast, elongation decreased progressively with increasing V content, dropping from 9.8% at 0% V to 4.0% at 0.56% V (a 59% reduction). Hardness increased modestly with V addition, rising from 217 HBW at 0% V to 249 HBW at 0.56% V.

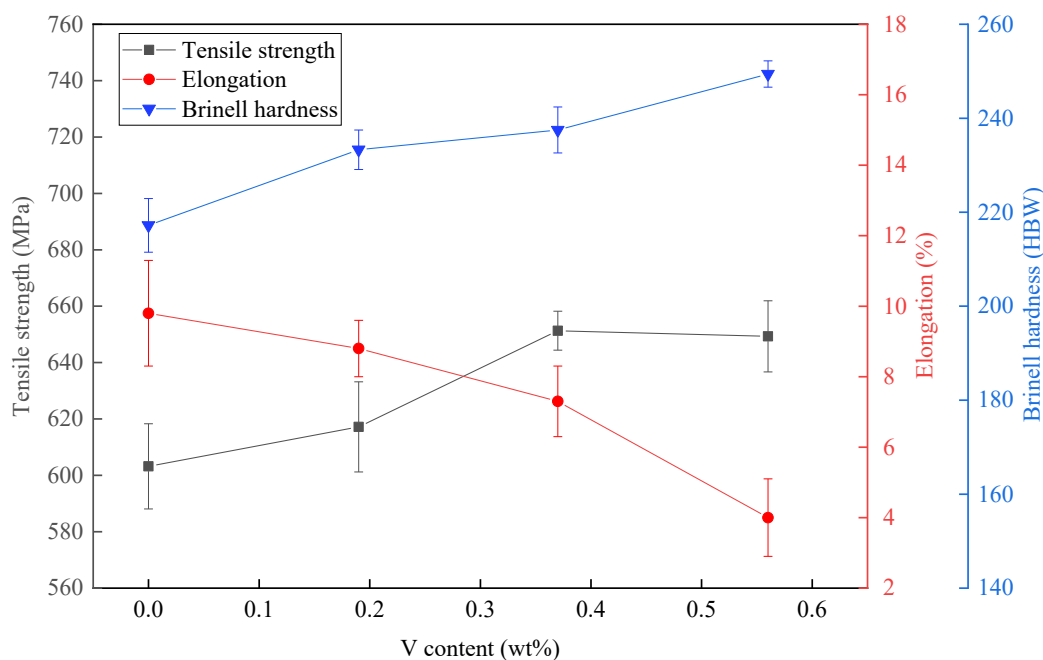


Figure 5. Room-temperature mechanical properties of Si–Mo ductile iron with varying V contents.

In Si–Mo ductile iron, V preferentially combines with C to form (V,Mo)C carbides. This suppresses the precipitation of continuous, fishbone-like M_6C carbides and transforms the carbide morphology from a fishbone-like network to dispersed blocky particles. This morphological refinement reduces stress concentration and impedes dislocation motion, thereby increasing strength. However, when the V content exceeds 0.37%, the volume fraction of (V,Mo)C carbides increases further. Although hardness continues to increase, the intrinsic brittleness of the carbides becomes more influential, partially offsetting the strengthening benefit; as a result, the strength increment saturates while elongation continues to decrease. In addition, the pearlite fraction decreases progressively with increasing V content, which further diminishes strength. When V increases from 0.37% to 0.56%, dispersion strengthening from (V,Mo)C carbides approaches saturation and can no longer compensate for the strength loss associated with the continued reduction in pearlite. Consequently, the tensile strength changes only slightly, from 651 to 649 MPa.

Figure 6 shows the SEM fractographs of the room-temperature tensile fracture surfaces of Si–Mo ductile iron with different V contents. As shown in Figure 6a, the V-free specimen exhibited a mixed ductile–brittle fracture mode, with brittle fracture features predominating. Numerous graphite nodules were exposed on the fracture surface. The matrix mainly showed cleavage and quasi-

cleavage fracture, characterized by distinct river patterns and fan-shaped cleavage facets. At higher magnification (Figure 6e), a small number of fine dimples were observed around graphite nodules and along tearing ridges. For the specimen containing 0.19% V, the fraction of cleavage facets increased and the number of dimples decreased. At V contents of 0.37% and 0.56%, the fracture surfaces were dominated by cleavage fracture, with predominantly flat cleavage facets and only a few tearing ridges. High-magnification observation (Figure 6f) further revealed typical brittle-fracture features, including river patterns and fan-shaped cleavage steps, indicating limited plastic deformation of the matrix.

V refines the matrix via the formation of dispersed carbides and by modifying carbide morphology, thereby increasing strength. However, when the V content exceeds 0.37%, the higher carbide fraction increases matrix brittleness, promoting a transition in fracture behavior from mixed ductile–brittle fracture to predominantly brittle cleavage fracture.

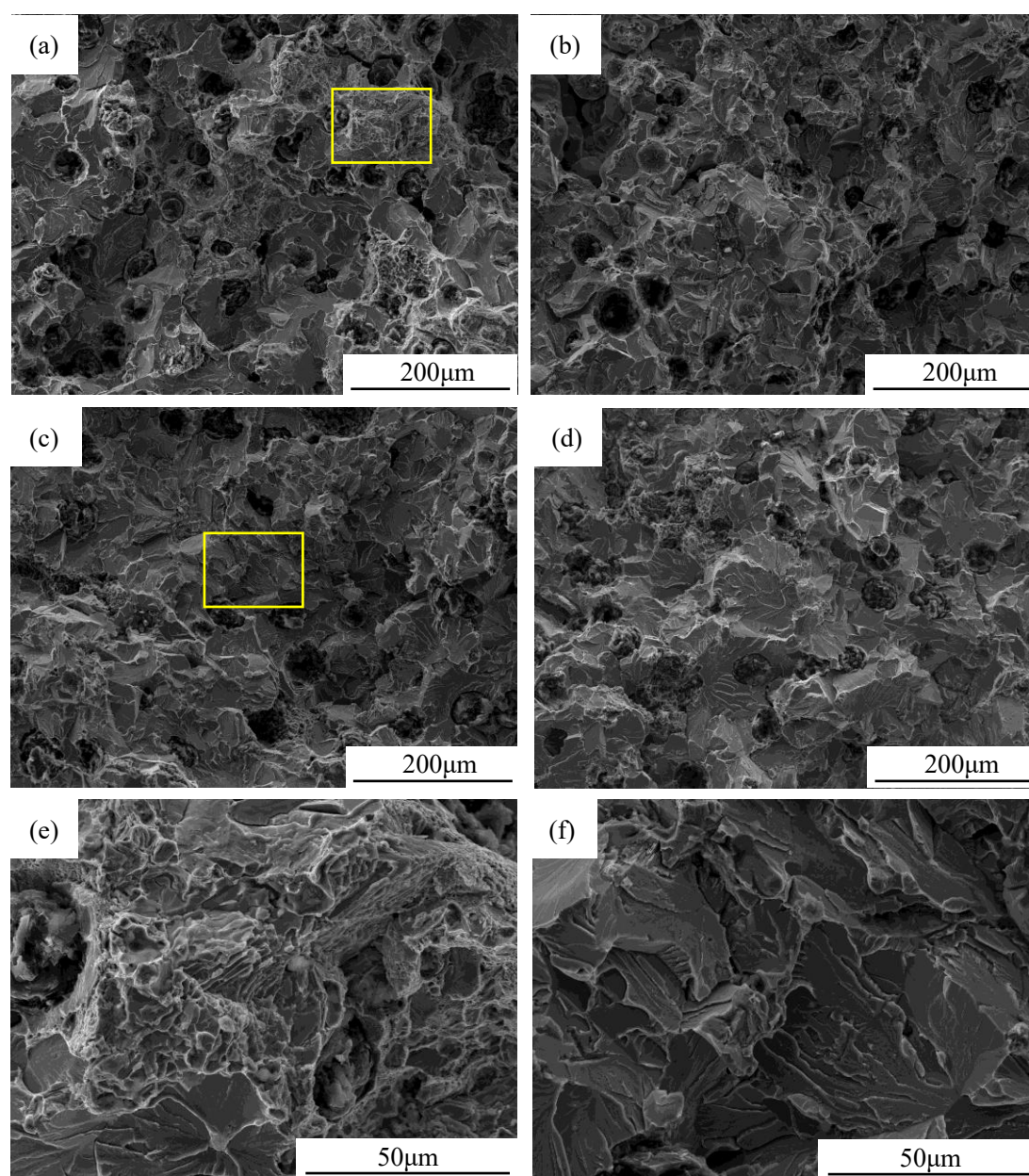


Figure 6. Room-temperature tensile fracture morphologies of Si-Mo ductile iron with

different V contents: (a) 0%, (b) 0.19%, (c) 0.37%, (d) 0.56%, (e) magnified view of the rectangular area in (a), and (f) magnified view of the rectangular area in (c).

High-Temperature Oxidation

High-temperature components, such as exhaust manifolds, experience not only mechanical loading at room temperature but also prolonged exposure to elevated temperatures up to 800 °C. Under these conditions, the material must resist high-temperature oxidation as well as creep and fatigue damage. It is therefore necessary to clarify how V influences the microstructure and properties of Si–Mo ductile iron in high-temperature environments.

Figure 7 shows the surface oxide-film morphologies of Si–Mo ductile iron specimens with different V contents after exposure at 800 °C for 60 h. In the V-free specimen, the oxide film was continuous, compact, and uniform in thickness. With the addition of 0.19% V, the oxide film thickened and pores began to form. In the specimen containing 0.37% V, the oxide film was substantially thicker and exhibited a loose, porous structure; localized internal oxidation extending from the surface into the matrix was also observed. In the 0.56% V specimen, the oxide layer thickened further, and dispersed internal oxidation was observed in regions farther from the surface.

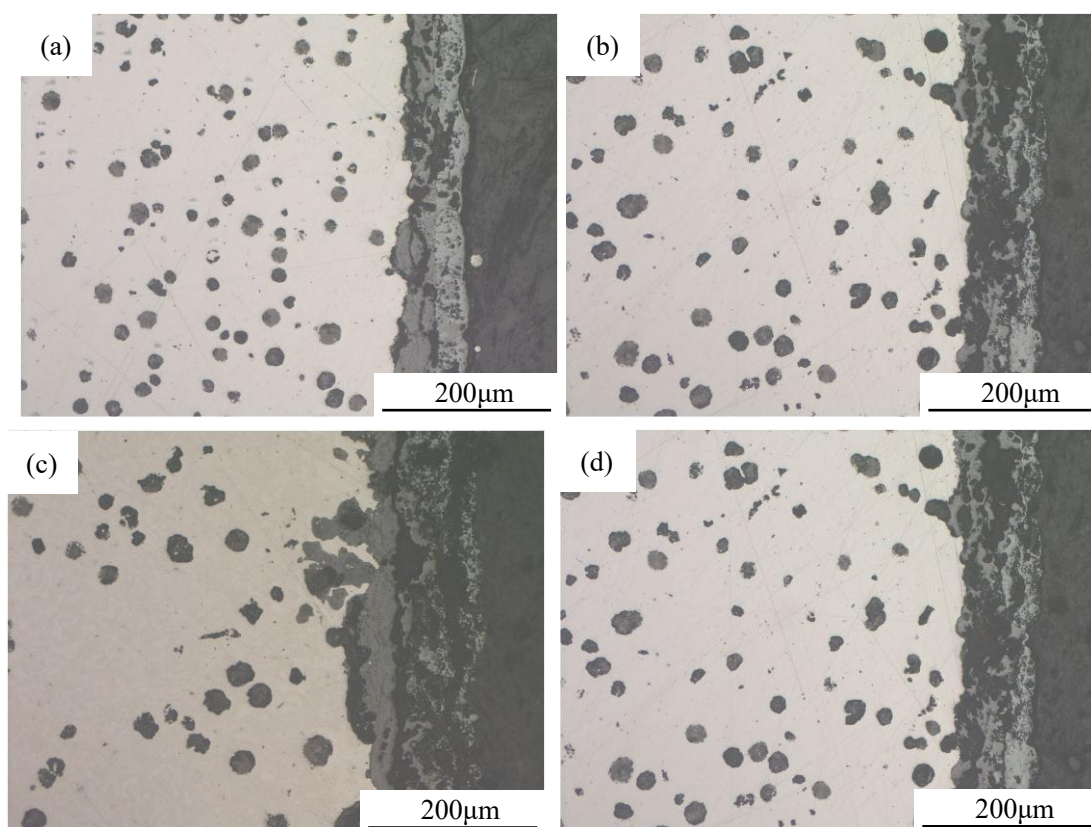
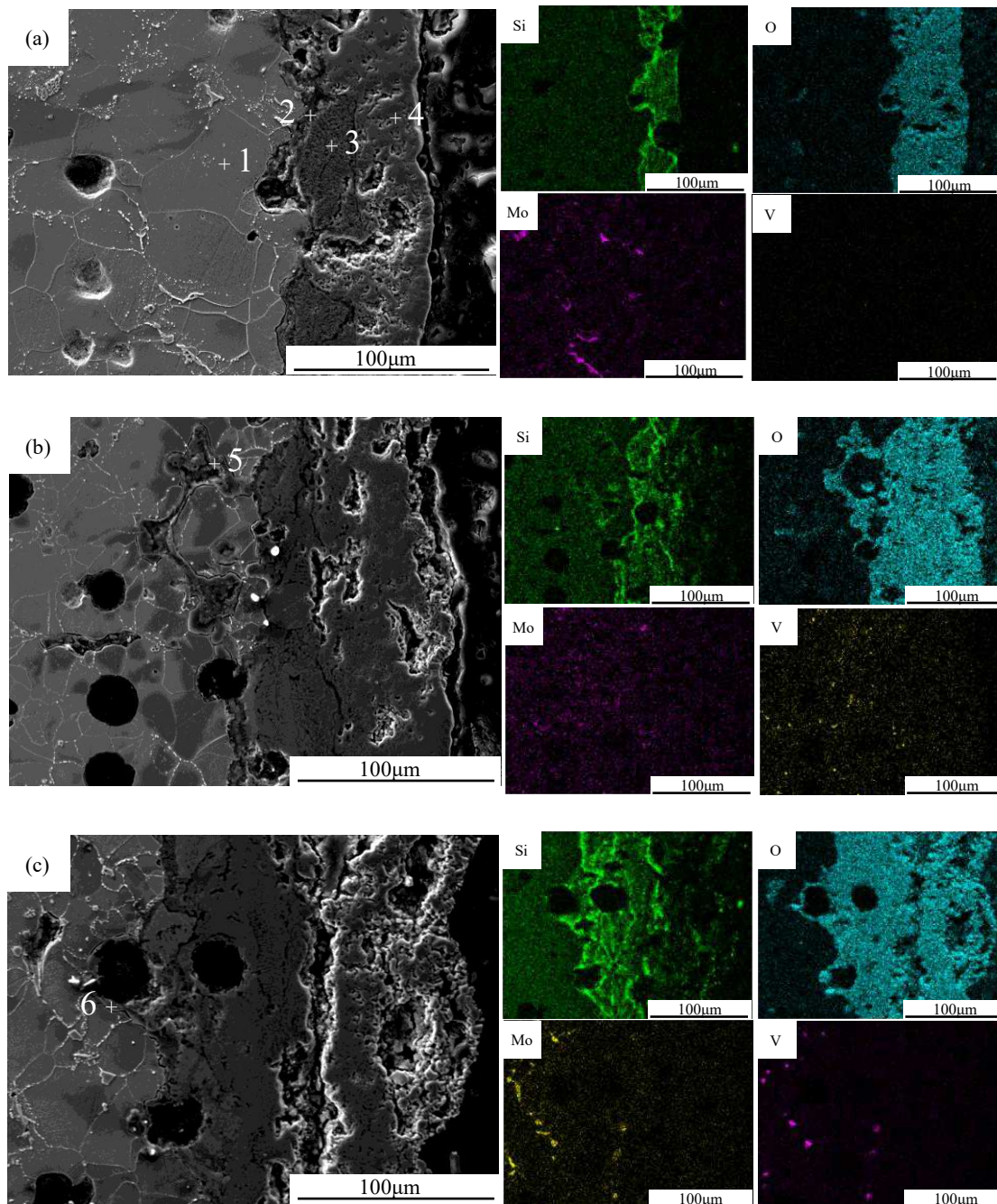


Figure 7. Surface oxide film morphology of Si–Mo ductile iron with different V contents: (a) 0%, (b) 0.19%, (c) 0.37%, and (d) 0.56%.

Figure 8 shows the surface oxide morphology and EDS results for Si–Mo ductile iron specimens with different V contents after oxidation at 800 °C for 60 h. In the V-free specimen, the oxide film was continuous and compact; the inner layer was rich in Si, whereas the outer layer

contained less Si. In the V-containing specimens, the inner Si-rich oxide layer remained relatively dense; however, the outer oxide layer became progressively looser with increasing V content, accompanied by a non-uniform Si distribution. Specifically, Si was enriched at the inner oxide/matrix interface and at the interface between the inner and outer oxide layers. In internally oxidized matrix regions, the Si content was higher than in the unoxidized matrix.



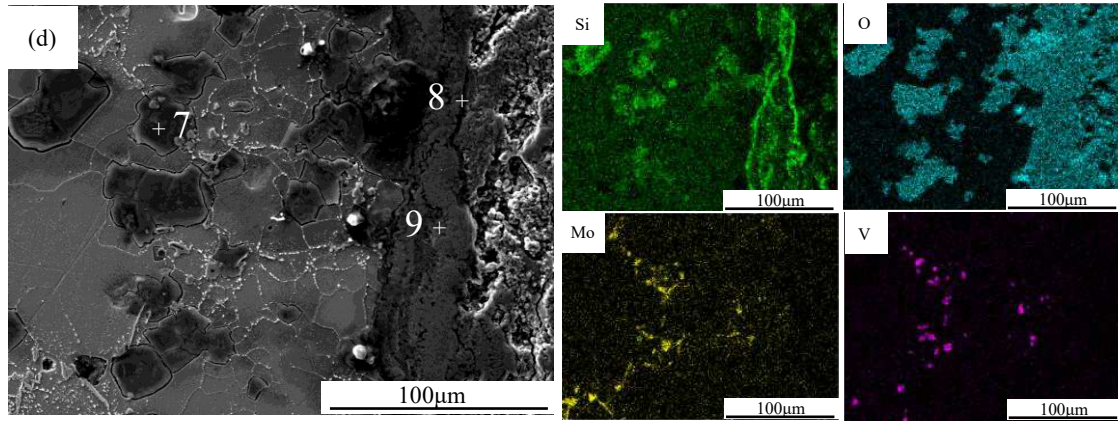


Figure 8. SEM images and EDS analysis of the surface oxide layer on Si-Mo ductile iron with varying V contents: (a) 0%, (b) 0.19%, (c) 0.37%, and (d) 0.56%.

EDS point analyses were performed in the regions marked in Figure 8 to determine the phase constitution and elemental distribution; the results are summarized in Table 3. In the V-free specimen, Point 1 was located in an Fe-rich region corresponding to the matrix. Point 2 was positioned at the matrix/oxide-film interface and contained high Si (31.23 at%) and O (51.94 at%), indicating that the interfacial oxide was predominantly SiO_2 . Point 3, located within the inner oxide layer, was enriched in Fe (50.45 at%) and O (32.62 at%), whereas the Si content (9.76 at%) was significantly lower than that at the interface. Point 4, located in the outer oxide layer, contained Fe (50.56 at%) and O (44.00 at%) with negligible Si (0.06 at%). These results indicate that the inner oxide layer comprised a complex Fe-Si oxide, whereas the outer layer consisted primarily of Fe oxides.

In the 0.19% V specimen, the oxide penetrated into the matrix. Point 5, located within this oxidized region, contained V (3.27 at%) and O (39.49 at%), indicating that V participated in the oxidation process. In the 0.37% V specimen, Point 6 was located near a graphite nodule at the oxidation interface and was enriched in Fe (31.80 at%) and O (35.52 at%), consistent with graphite-assisted oxidation. In the 0.56% V specimen, Point 7 was situated in an internally oxidized region of the matrix; the O and Si contents reached 51.82 at% and 14.96 at%, respectively, with the Si content being markedly higher than in the unoxidized matrix at Point 1. Point 8 was located at the interface between the inner and outer oxide layers and exhibited a Si content of 15.97 at%, which was significantly higher than that at Point 9 (4.40 at%), located away from the interface.

Table 3. EDS analysis results of the points marked in Figure 8

Point	C(at%)	Si(at%)	Fe(at%)	O(at%)	Mo(at%)	V(at%)
1	5.93	6.36	82.63	4.12	0.11	-
2	7.98	31.23	8.11	51.94	0.17	-
3	6.91	9.76	32.62	50.45	0.18	-
4	5.20	0.06	50.56	44.00	0.14	-
5	7.29	5.83	40.87	39.49	0.26	3.27
6	19.46	10.72	31.80	35.52	0.40	0.18
7	7.98	14.96	23.99	51.82	0.62	0.44

8	10.11	15.97	25.48	47.66	0.20	0.11
9	5.67	4.40	47.57	41.31	0.61	0.34

During the initial stage of oxidation of Si–Mo ductile iron, Fe reacts preferentially with O because of its higher diffusion rate. Consequently, an outer $\text{Fe}_2\text{O}_3/\text{Fe}_3\text{O}_4$ layer forms rapidly on the surface. With continued exposure, Si enriches at the metal/oxide interface and oxidizes to form a dense Fe_2SiO_4 intermediate layer; a very thin SiO_2 -rich layer also develops at the interface [35]. This continuous Si-rich oxide layer impedes the inward diffusion of O and the outward diffusion of metal ions, thereby significantly reducing the oxidation rate.

V addition degrades the oxidation resistance of Si–Mo ductile iron. Because V has a strong affinity for O, it can form several oxides (e.g., V_2O_5 , VO_2 , and V_2O_3); under high-temperature oxidation conditions, V_2O_5 is the most stable. V_2O_5 melts at approximately 690 °C and is therefore liquid at high temperatures [36]. Molten V_2O_5 can disrupt the previously formed protective SiO_2 layer and promote the formation of multicomponent complex oxides [37]. Owing to its high fluidity and permeability, liquid V_2O_5 can infiltrate the material along cracks, pores, and grain boundaries, transporting O into the interior and creating favorable conditions for internal oxidation. Furthermore, the presence of liquid V_2O_5 provides fast diffusion pathways for O and metal ions, leading to a pronounced increase in the oxidation rate.

Microstructural Evolution at High Temperature

In addition to surface oxidation, Si–Mo ductile iron undergoes internal microstructural changes during prolonged high-temperature exposure. To elucidate this evolution under high-temperature conditions, DSC analysis was performed; the results are shown in Figure 9.

The DSC curves reveal that all specimens exhibited an endothermic peak at 720 °C during heating. At elevated temperatures, lamellar pearlite decomposes into granular pearlite [38]; this peak therefore corresponds to the pearlite decomposition temperature. With further heating to approximately 890 °C, another peak appeared in all specimens. Because Si–Mo ductile iron has a high Si content, the eutectoid transformation temperature is increased; accordingly, this peak is attributed to the eutectoid transformation. By contrast, Si does not significantly affect the eutectic temperature, and a distinct peak at 1160 °C was observed, corresponding to the eutectic temperature. During austenitization heating (890–1160 °C), additional endothermic features were observed. The V-free specimen exhibited a single peak at approximately 980 °C, whereas the V-containing specimen showed an additional weak peak near 1050 °C. Previous studies indicate that MC carbides have higher thermal stability and higher dissolution temperatures than M_6C carbides during austenitization [39,40]. Accordingly, the peak at 980 °C is assigned to $(\text{Mo,Fe,Si})_6\text{C}$ dissolution, and the peak at 1050 °C to $(\text{V,Mo})\text{C}$ dissolution.

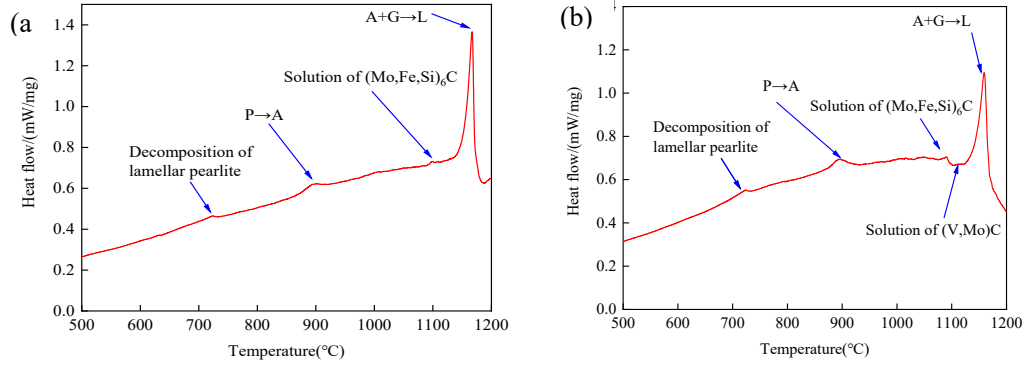
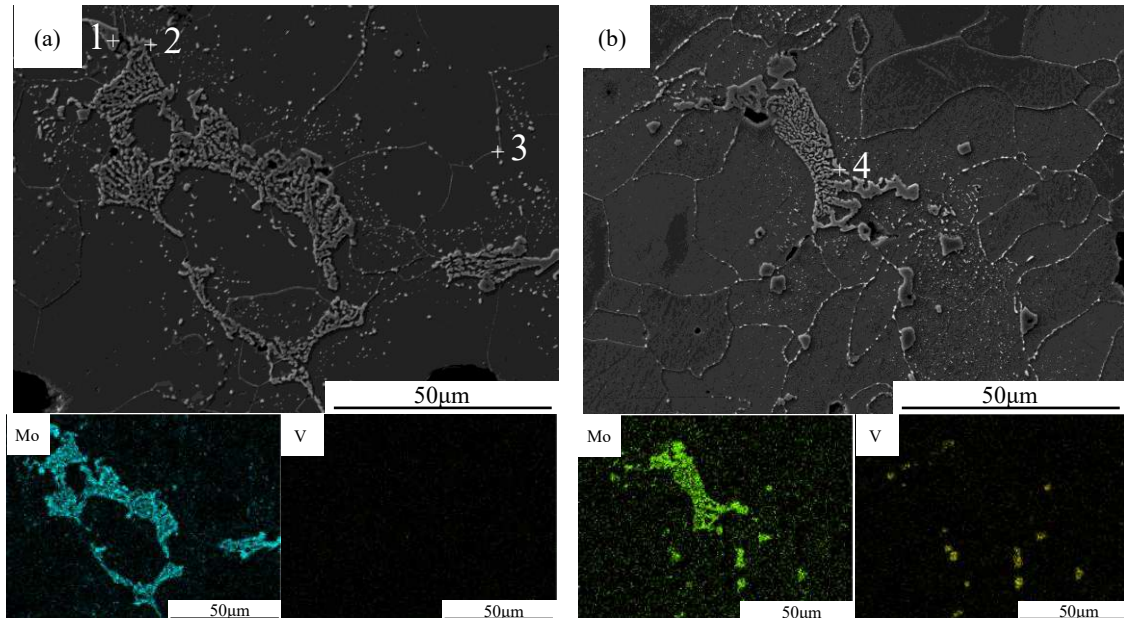


Figure 9. DSC curve of Si–Mo ductile iron during heating: (a) 0% V and (b) 0.37% V.

After DSC analysis, the microstructure following long-term high-temperature exposure was examined to further elucidate the effect of V on microstructural evolution. Figure 10 shows the microstructural morphologies of Si–Mo ductile iron with different V contents after holding at 800 °C for 60 h. As shown in Figure 10a, the V-free specimen exhibited blunting and partial spheroidization at the edges of the fishbone-like carbides relative to the room-temperature microstructure. The lamellar pearlite decomposed into granular pearlite, and numerous fine particles precipitated along grain boundaries and within grains. In the V-containing specimens, the microstructural changes were less pronounced than in the V-free alloy. The V-rich blocky carbides underwent further spheroidization, the number of strip-like carbides decreased, and a greater number of fine granular carbides appeared at grain boundaries and within grains.



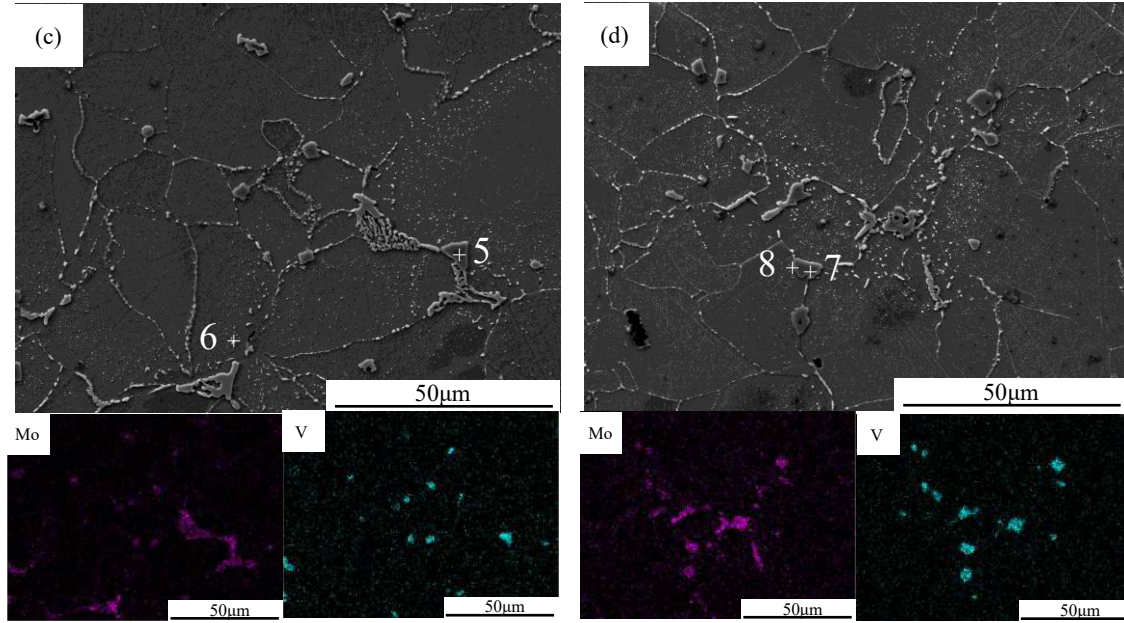


Figure 10. Carbide morphology and EDS analysis of Si-Mo ductile iron after high-temperature exposure: (a) 0%, (b) 0.19%, (c) 0.37%, and (d) 0.56%.

Table 4 shows the EDS results for the regions marked in Figure 10. In the V-free specimen, Points 1 and 2 were located at the center and edge of the network-like $(\text{Mo,Fe,Si})_6\text{C}$ carbide, respectively. The alloying-element distributions became more uniform at the center and edge regions. Point 3 was located in a granular carbide at a grain boundary. This region was Fe-rich and contained 21.15 at% C and 9.85 at% Mo, indicating the precipitation of Mo-rich carbides at grain boundaries during prolonged high-temperature exposure. Points 4 and 5 corresponded to the $(\text{Mo,Fe,Si})_6\text{C}$ and $(\text{V,Mo})\text{C}$ carbides, respectively. Their chemical compositions did not differ significantly from those observed in the room-temperature microstructure. Point 6 was located in a granular carbide precipitated within a grain. This point contained a trace amount of V (0.72 at%), whereas the concentrations of the other elements were similar to those in the grain-boundary carbide (Point 2). Points 7 and 8 were located at the center and edge of a $(\text{V,Mo})\text{C}$ carbide, respectively. Unlike the room-temperature microstructure, no pronounced difference in V content was observed between the center and edge regions.

Table 4. EDS analysis results of the points marked in Figure 10

Point	C(at%)	Si(at%)	Fe(at%)	Mo(at%)	V(at%)
1	22.53	11.09	42.82	22.90	-
2	24.08	10.84	43.60	20.97	-
3	21.15	9.44	59.39	9.85	-
4	25.81	11.21	39.14	20.25	1.32
5	42.30	0.12	2.94	11.39	43.25
6	27.37	9.55	51.33	11.03	0.72
7	43.99	0.15	2.61	12.43	40.79
8	47.67	0.28	3.44	10.01	38.82

M₆C and MC carbides exhibit high thermal stability at elevated temperatures, whereas pearlite decomposes under these conditions. The tips of fishbone-like M₆C carbides have high interfacial energy; at high temperatures, enhanced atomic diffusion promotes dissolution at these regions into the matrix. The dissolved species subsequently redeposit on flatter carbide surfaces, resulting in coarsening and blunting of sharp features. Concurrently, diffusion of C, Mo, and Fe between the carbides and the matrix reduces compositional gradients at the interface, further lowering interfacial energy and promoting edge rounding. For MC-type carbides, V and Mo segregate from the supersaturated ferrite matrix to existing MC particles and progressively incorporate into the MC lattice. This process leads to MC coarsening and gradual homogenization of the composition between the particle interior and periphery.

High-Temperature Mechanical Properties and Fracture Morphology

Figure 11 shows the high-temperature tensile properties of Si–Mo ductile iron with different V contents measured at 800 °C. As the V content increased from 0% to 0.56%, the tensile strength increased monotonically, whereas elongation decreased steadily. The V-free specimen exhibited a tensile strength of 48.7 MPa and an elongation of 48.0%. By contrast, the 0.56% V specimen reached 59.7 MPa with an elongation of 40.7%, corresponding to an ~23% increase in strength and an ~15% decrease in elongation relative to the V-free alloy.

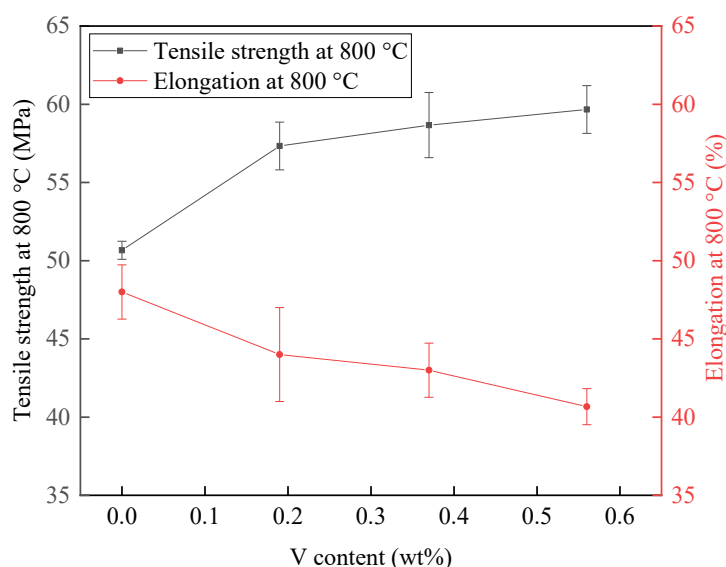


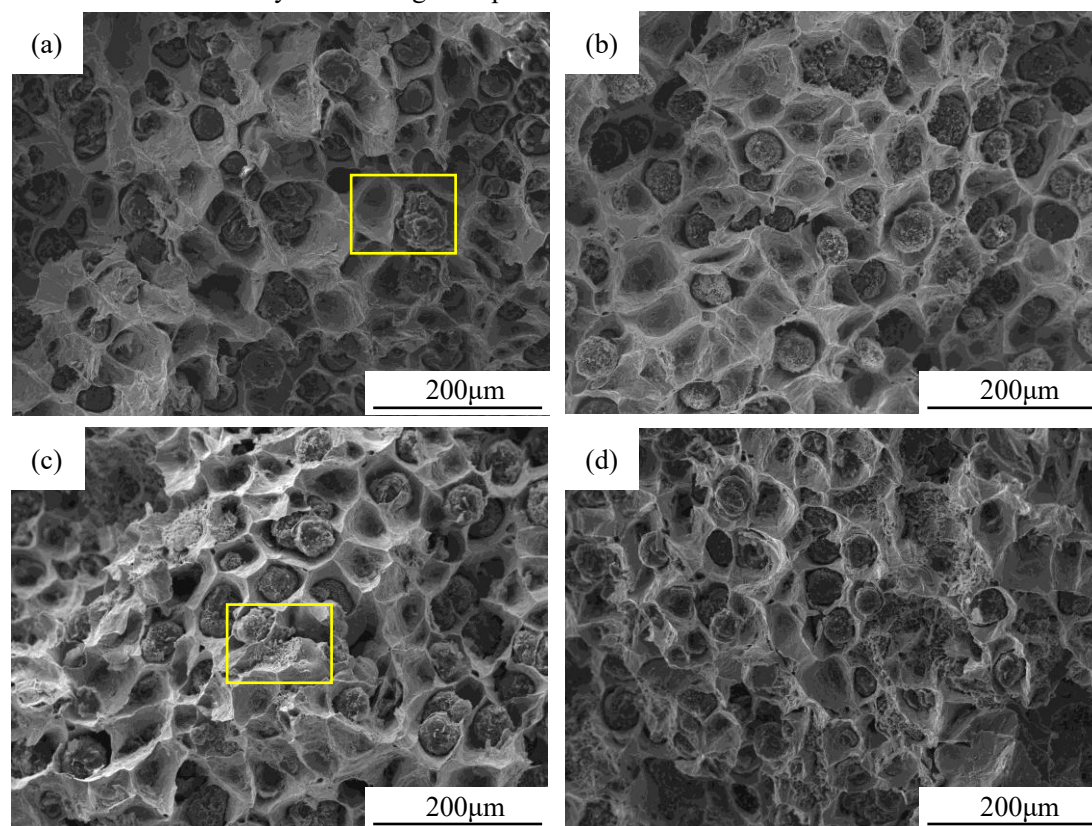
Figure 11. High-temperature mechanical properties of Si–Mo ductile iron with different V contents.

At room temperature, the strength peaked at 0.37% V, whereas at 800 °C it increased continuously up to 0.56% V. Moreover, V provided a more pronounced strengthening effect at high temperature. At elevated temperatures, the matrix softens and the overall plasticity increases; meanwhile, dispersed MC-type carbides remain thermally stable and can effectively pin grain boundaries. At room temperature, when the V content exceeds 0.37%, the increased carbide fraction promotes brittle fracture and reduces the pearlite content, limiting further gains in strength. Under high-temperature conditions, pearlite has already decomposed, and the additional MC carbides formed at higher V contents continue to impede matrix softening. Consequently, carbide strengthening is more fully retained at high temperature, leading to a continuous increase in strength

with increasing V content.

In addition, the detrimental effect of V on elongation was much less pronounced at elevated temperature than at room temperature. Compared with the V-free specimen, the addition of 0.56% V reduced the room-temperature elongation by 59%, whereas the elongation at high temperature decreased by only ~15%. This behavior is attributed to the enhanced plastic deformability of the matrix at elevated temperatures. Although dispersed MC carbides increase deformation resistance, they do not induce severe stress concentrations. Consequently, the alloy maintains good high-temperature ductility while achieving increased strength.

Figure 12 shows the fracture morphologies of Si–Mo ductile iron with different V contents after tensile testing at 800 °C. Compared with the room-temperature fracture surfaces, the high-temperature surfaces were covered by an oxide layer and exhibited numerous dimples. In the V-free specimen, the fracture surface exhibited pronounced plastic deformation, with relatively deep dimples, indicating a typical high-temperature ductile-fracture morphology. High-magnification imaging (Figure 12e) revealed that interfacial separation frequently occurred at the interface between graphite nodules and the matrix as a result of high-temperature softening, leading to cavity formation. With increasing V content, the dimple depth gradually decreased. High-magnification observation of the 0.37% V specimen (Figure 12f) revealed intergranular fracture in the vicinity of carbide particles. The fracture surface suggests that the carbides largely retained their original morphology at high temperature, whereas the surrounding matrix underwent significant plastic deformation due to softening. These observations confirm the high thermal stability of MC-type carbides and their ability to resist high-temperature deformation.



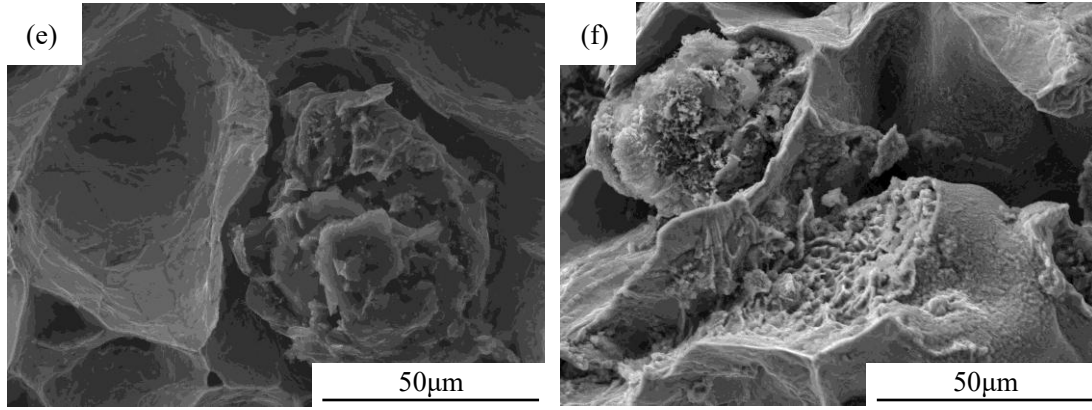


Figure 12. High-temperature fracture morphology of Si-Mo ductile iron with different V contents: (a) 0%, (b) 0.19%, (c) 0.37%, (d) 0.56%, (e) rectangular area in (c), and (f) rectangular area in (d).

Figure 13 presents the high-temperature creep-rupture behavior of Si-Mo ductile iron specimens with different V contents tested at 800 °C under a constant tensile stress of 25 MPa. The V-free specimen exhibited a creep-rupture life of 0.9 h. With increasing V content, the rupture life increased markedly: 5.9 h at 0.19% V, 8.7 h at 0.37% V, and 9.7 h at 0.56% V. Compared with the V-free condition, 0.56% V yielded an approximately 10-fold increase in creep-rupture life.

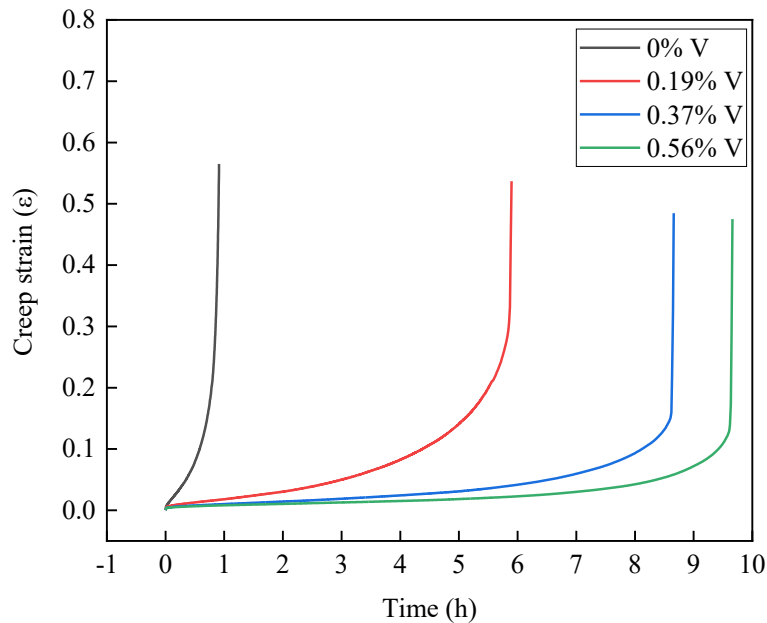


Figure 13. High-temperature creep-rupture properties of Si-Mo ductile iron with different V contents.

Failure during long-term exposure at 800 °C under stress is dominated by grain-boundary sliding and the accumulation of creep cavities. V promotes the formation of dispersed MC carbides that effectively pin grain boundaries while avoiding the development of a continuous carbide network. As a result, creep-crack initiation and propagation are significantly delayed. Furthermore, the high stability of MC particles during prolonged exposure enables sustained suppression of

matrix softening and improved creep resistance. These effects are most evident under creep-rupture conditions; accordingly, the increase in rupture life with V content greatly exceeds the corresponding improvement in short-term tensile strength.

Figure 14 shows the fatigue cycles of pre-oxidized Si–Mo ductile iron specimens with different V contents tested at 700 °C under an applied stress of 50 MPa. As the V content increased, the fatigue life first increased and then decreased. The V-free specimen exhibited a fatigue life of 5.68×10^5 cycles. Increasing V to 0.37% increased fatigue life to a maximum of 1.278×10^6 cycles, approximately 1.3 times that of the V-free specimen. With a further increase to 0.56% V, the fatigue life decreased to 1.140×10^6 cycles.

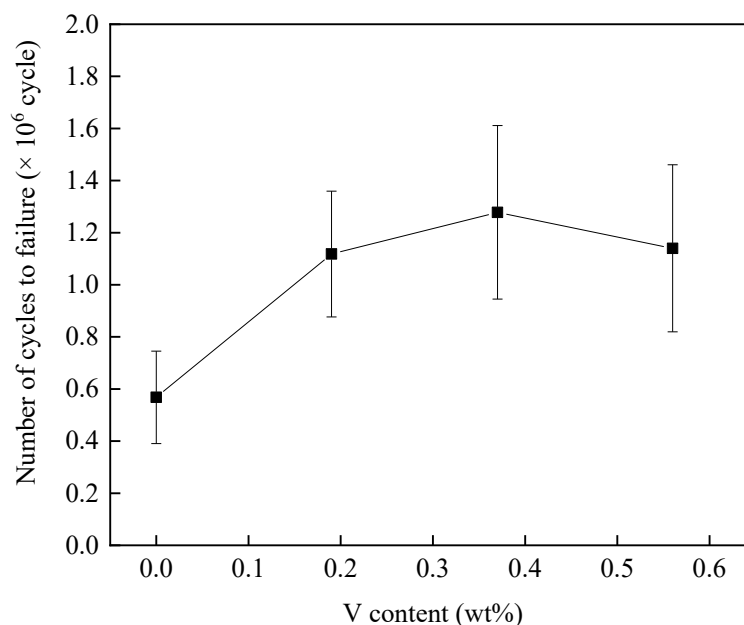


Figure 14. Pre-oxidation high-temperature fatigue life of Si–Mo ductile iron with different V contents.

With increasing V content, the oxidation resistance progressively deteriorated, resulting in a thicker oxide film. As V increased from 0% to 0.37%, the fatigue life increased continuously, primarily because the enhancement in high-temperature strength offset the adverse effect of oxidation. When V was further increased to 0.56%, the high-temperature strength continued to increase; however, the associated loss in oxidation resistance became pronounced. The oxide film became excessively thick and poorly adherent, and substantial internal oxidation occurred. Under these conditions, the negative effect of oxidation on fatigue life outweighed the benefit of higher strength, leading to a reduction in fatigue cycles. In addition, the elastic modulus mismatch between the carbides and the matrix promotes crack initiation at the carbide/matrix interface under high-temperature cyclic loading, followed by rapid crack propagation and fracture. Consequently, fatigue life does not increase monotonically with V content and instead exhibits a peak at intermediate V levels.

Conclusion

The effects of V content on the microstructure, room-temperature tensile properties, high-temperature oxidation behavior, high-temperature tensile properties, high-temperature creep-

rupture performance, and pre-oxidation high-temperature fatigue performance of Si–Mo ductile iron were investigated. The main conclusions are as follows:

(1) With increasing V content, the pearlite fraction in the matrix decreased continuously. V addition promoted the formation of (V,Mo)C carbides and inhibited the continuous precipitation of fishbone-like (Mo,Fe,Si)₆C carbides. Accordingly, the carbide morphology shifted from a continuous fishbone-like network to dispersed blocky particles with a more uniform distribution.

(2) At 800 °C, both (V,Mo)C and (Mo,Fe,Si)₆C carbides showed coarsening accompanied by edge blunting and spheroidization. As V content increased, the oxide film became thicker and less compact, and internal oxidation was observed.

(3) The room-temperature tensile strength increased initially and then decreased with increasing V content, reaching a maximum of 651 MPa at 0.37% V—approximately 8% higher than that of the V-free specimen (604 MPa). In contrast, the high-temperature tensile strength at 800 °C increased monotonically with V addition, rising from 48.7 MPa for the V-free specimen to 59.7 MPa at 0.56% V, representing an approximately 23% improvement.

(4) V significantly enhanced the creep resistance of the material. The creep-rupture life at 800 °C under 25 MPa increased from 0.9 h for the V-free specimen to 9.7 h at 0.56% V, representing an approximately 10-fold enhancement. The pre-oxidation high-temperature fatigue life at 700 °C increased initially and then decreased with increasing V content, peaking at 1.278×10^6 cycles at 0.37% V, which was approximately 1.3 times that of the V-free specimen.

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Author Contributions

Tong Jin: Writing – review & editing, Writing – original draft, Methodology. Qizhou Cai: Writing – review & editing, Supervision. Xu Chen: Methodology, Formal analysis. Yuanbao Zheng: Investigation. Lujun Jiang: Writing – original draft. Song Xuanyi: Writing – review & editing. Fuan Yuan: Validation.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that may have influenced the work reported in this paper.

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