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A study of laves phase in high Nb non-oriented silicon steel for electric automobiles

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Abstract

In the study of precipitates in high-Nb non-oriented silicon steel for the rotor of electric automobiles, it was found that mass dispersed Nb-rich precipitates were formed during the rolling process of the steel, these precipitates are presumed to be Laves phases in published literatures. In this regard, two types of Laves phases of high-Nb non-oriented silicon steel for electric automobiles and their crystallographic relationship with the Fe matrix were determined using SAED, FFT and IFFT, and calculation of steel strength enhancement by precipitation strengthening. The results showed the elliptical Laves phase is Fe16Nb6Si7 and the rectangular strip Laves phase is Nb7P4, and the orientation relationship between Fe16Nb6Si7 phase and Fe matrix is : [001]Fe16Nb6Si7∥[001]Fe, [101]Fe16Nb6Si7∥[110]Fe, [113]Fe16Nb6Si7∥[112]Fe, the orientation relationship between Nb7P4 phase and Fe matrix is : [130]Nb7P4∥[001]Fe, [102]Nb7P4∥[110]Fe, [110]Nb7P4∥[112]Fe; Both Fe16Nb6Si7 and Nb7P4 precipitates have a good coherent relationship with the Fe matrix; The calculation results show that Fe16Nb6Si7, Nb7P4 and other precipitates can improve the yield strength of non-oriented silicon steel, but at the same time, the magnetic properties of the steel are expected to be negatively impacted. The research results can provide important theoretical guidance on the influence of thermo-mechanical processing on balancing the mechanical and magnetic properties of high niobium non-oriented silicon steel for electric vehicle rotor.

1. Introduction

Due to the continuous deterioration of the environment, traffic has become synonymous with air pollution, electric automobiles have become the development trend of the world’s automobile industry [1–4]. As one of the important components of electric automobiles, the driver motor rotor system plays an important role, and the use of non-oriented electrical steel is the guarantee for high efficiency, high power density vehicle motors [5]. Due to the high speed of the electric automobiles drive motor and high torque requirements when starting and accelerating, non-oriented silicon steel for the rotor is required to have excellent mechanical properties while meeting high magnetic induction intensity and low iron loss [6–8]. At present, the methods to improve the strength of non-oriented electrical steel mainly include solid solution strengthening, precipitation strengthening, fine grain strengthening and dislocation strengthening [9–11]. It was pointed out that the mechanical properties of non-oriented silicon steel can be greatly improved by adding appropriate amount of Ni element without seriously deteriorating the magnetic induction strength and iron loss [12]. However, the addition of expensive Ni increases the production cost. Microalloying of engineering structural steel with Nb and other elements can achieve the purpose of both high strength and low cost [13–15], but in the study of high-Nb non-oriented silicon steel, it was found that mass dispersed Nb-rich precipitates were formed during the rolling process of this component steel [16, 17], the strength of non-oriented silicon steel was slightly improved by precipitation strengthening, but magnetic properties were also sacrificed, they speculated that these dispersed Nb-rich precipitates may be Laves phase, but it still needs to be characterized by the structure of the phase.
In this study, the types of the Nb-rich precipitates and their coherent relationship with the matrix are determined by the overlapped selected area electron diffraction (SAED) patterns and the fast Fourier transform (FFT) of high-resolution (HR) TEM micrograph and the inverse fast Fourier transform (IFFT) patterns, and the effects of Nb-rich precipitates on the mechanical and magnetic properties of non-oriented silicon steel were simply calculated, which provides a theoretical basis for the precipitation control of non-oriented silicon steel for the production of electric vehicle drive motor rotor.

2. Materials and methods

25 kg ingot was produced by vacuum induction melting and its chemical composition is shown in table 1. Figure 1 is the thermodynamic calculation diagram of phase equilibrium content at different temperatures according to the composition of the non-oriented silicon steel, which was calculated by Thermo-Calc software (based on TCFe9 database). It is shown in the figure that MnS, NbC, AlN, FeNbP, NbP and Laves phases may be precipitated in the composition system of this steel. According to the thermodynamic calculation results, NbP precipitates begin to dissolve at 400 °C, the content of Laves precipitates reached a peak at 680 °C, and then gradually redissolved.

The hot rolling process of non-oriented silicon steel is developed based on thermodynamic calculation, and the rolling process is shown in table 2. The initial rolling temperature of the slab was 1250 °C, because according to thermodynamic calculation at this temperature, most precipitates in the steel have been dissolved in the steel. Hot rolling was performed with 33 mm thick plates to 2.5 mm via five passes, and the final rolling temperature was 750 °C. Next, the hot-rolled sample was simulated coiled at 650 °C for 2 h and then cooled with the furnace.

The JEM-2100F transmission electron microscope (TEM) equipped with energy-dispersive X-ray spectroscopy analyzer (EDS), operated at an acceleration voltage of 200 kV, was used to analyze the interfaces between adjacent phases in different precipitates, The high-resolution TEM (HR-TEM) images were post-processed by fast Fourier transforming (FFT) and inverse fast Fourier transforming (IFFT) using the Gatan Digital Micrograph™ to treatment. TEM thin foil was prepared by using MODEL-110 double jet electrolytic polishing instrument.

3. Results and discussion

Figure 2 shows the precipitates observed by TEM in the as-cast strip, the types of precipitates that were found by using the carbon extraction replica method, the content of Si and Fe in the replicas samples is due to the high

| C  | Si  | Mn  | Alt | N   | P   | S   | Nb  | Ce |
|----|-----|-----|-----|-----|-----|-----|-----|----|
| ≤0.003 | 3.0 | 0.2 | 0.8 | ≤0.003 | 0.010 | ≤0.003 | 0.15 | 15 ppm |

Table 1. Composition analysis of experimental steels (wt%).
Table 2. Rolling experiment scheme.

| Initial Rolling Temperature / °C | First Pass | Second Pass | Third Pass | Fourth Pass | Fifth Pass | Final Rolling Temperature / °C |
|---------------------------------|------------|-------------|------------|-------------|------------|-----------------------------|
|                                 | Degree Of Reduction / % | Thickness / mm | Degree Of Reduction / % | Thickness / mm | Degree Of Reduction / % | Thickness / mm | Degree Of Reduction / % | Thickness / mm | Degree Of Reduction / % | Thickness / mm | Degree Of Reduction / % | Thickness / mm |
| 1250                            | 55         | 15          | 50         | 7.5         | 40         | 4.5           | 30         | 3.0         | 20         | 2.5         | 750         |               |
content of Si and Fe in the non-oriented silicon steel, which cannot be completely removed by hydrochloric acid and alcohol in the sample preparation process. In figure 2(a), it can be seen that the precipitated phase aggregate growth, the shape is mainly round, and the dominant size of precipitates was in the range of 27–46 nm; EDS spectra in figure 2(b) show that the precipitates were NbC.

Figure 3 shows precipitated phases of hot-rolled film samples observed by transmission electron microscopy. In figure 3(a), it can be seen that the shape of the precipitated phase is divided into elliptical and rectangular, and the number of precipitated phases was large and the size was small, the particle size of the elliptical precipitated phase was in the range of 31–49 nm, and the particle size of the rectangular precipitated phase was in the range of 39–76 nm. The precipitated phase was distributed and aggregated linearly and
regularly. EDS analysis in figures 3(b), (c) shows that elliptical precipitates are rich in Fe, Si, Nb and C, while rectangular precipitates are rich in Fe, Si, Nb, C and P. In order to further explore the type of precipitated phase in steel and its coherent relationship with the matrix, three electron diffraction spots with different crystal band axes were calibrated on the sample. The type of precipitated phase was determined by comparing the IFFT pattern. At the same time, the precipitated phase was photographed by high-resolution photography. The electron diffraction spots were compared by Fourier transform to ensure the accuracy of the type of precipitated phase.

The crystallographic relationship between the precipitated phase and the matrix was determined by inverse Fourier transform. The FFT also proves the correctness of electron diffraction calibration. Due to a large number of stacking faults and dislocations in rectangular precipitates, the selected area electron diffraction cannot be photographed. However, the high-resolution photographs of rectangular precipitates were calibrated by Fourier transform and comparing the PDF card.

By calibrating the selected area electron diffraction (figures 4–6(b)) and comparing the PDF card (107097-ICSD), it can be determined that the elliptical precipitates are Fe16Nb6Si7, and the Fe16Nb6Si7 phase space group with face-centered cubic structure is Fm-3 m. The lattice constants are a = b = c = 11.334, α = β = γ = 90°. The orientation relationship between Fe16Nb6Si7 phase and Fe matrix is: [001]Fe16Nb6Si7 || [001]Fe, [101]Fe16Nb6Si7 || [110]Fe, [110]Fe16Nb6Si7 || [112]Fe. High-resolution photo Fourier transform also proves the correctness of electron diffraction calibration. Due to a large number of stacking faults and dislocations in rectangular precipitates, the selected area electron diffraction cannot be photographed. However, the high-resolution photographs of rectangular precipitates were calibrated by Fourier transform (figures 4–6(c)) combined with EDS spectrum analysis. At the same time, compared with the PDF card (41238-ICSD), it was determined that the rectangular precipitates were Nb7P4, and the spatial group structure of Nb7P4 phase with monoclinic bottom structure was C12-m1. The lattice constant was a = 14.9503, b = 3.1398, c = 13.8478, α = γ = 90°, β = 104.743°, the orientation relationship between Nb7P4 phase and Fe matrix is: [130]Nb7P4 || [001]Fe, [102]Nb7P4 || [110]Fe, [110]Nb7P4 || [112]Fe.

Figure 4(a) shows that Fe16Nb6Si7 and Nb7P4 gather and grow together in a shape similar to ‘key’. The overlapped selected area electron diffraction (SAED) patterns taken on the Fe / Fe16Nb6Si7 interface (figure 4(b)) and the fast Fourier transform (FFT) of high-resolution (HR) TEM micrograph (figure 4(c), (d)) and the corresponding inverse fast Fourier transform (IFFT) pattern (figures 4(f), (g)) show parallel crystallographic axes of these two crystals: [101]Fe16Nb6Si7 || [110]Fe. The orientation relationship of Fe16Nb6Si7 and Fe can be expressed as (111)Fe16Nb6Si7 || (111)Fe and the intergranular space were 0.70 nm and 0.17 nm. The IFFT pattern (figure 4(i)) and the corresponding overlapped HR-TEM, FFT (inset) evidence the highly coherent interface between the Fe16Nb6Si7 and the Fe matrix. The FFT of the HR-TEM micrograph (figures 4(d), (e)) and the corresponding inverse fast Fourier transform (IFFT) pattern (figures 4(g), (h)) show parallel crystallographic axes of the Fe and Nb7P4 crystals: [102]Nb7P4 || [110]Fe. The orientation relationship of Nb7P4 and Fe can be expressed as (201)Nb7P4 || (111)Fe and the intergranular space were 0.72 nm and 0.17 nm, (020)Nb7P4 || (220)Fe.

Figure 4. Crystallography in the Fe / Fe16Nb6Si7 / Nb7P4 system along [110]Fe, [101]Fe16Nb6Si7, [102]Nb7P4 direction. (a) HR-TEM image and the corresponding FFT pattern (c–e) and the corresponding IFFT pattern (f, h); (b) SAED patterns taken at the Fe / Fe16Nb6Si7 interface; and IFFT pattern showing a marginal local lattice strain across the Fe / Fe16Nb6Si7 interface (i);
and the intergranular space were 0.19 nm and 0.21 nm, figure 4(j) evidence the highly coherent interface between the and Nb₇P₄ the Fe matrix.

Figure 5(a) shows that Fe₁₆Nb₆Si₇ and Nb₇P₄ gather and grow together in a shape similar to ‘hoe’; The overlapped SAED patterns taken on the Fe / Fe₁₆Nb₆Si₇ interface (figure 5(b)) and the FFT of HR-TEM micrograph (figures 5(c), (d)) and the corresponding IFFT pattern (figures 5(e), (g)) show parallel crystallographic axes of these two crystals: [001]Fe₁₆Nb₆Si₇ // [001]Fe, The orientation relationship of Fe₁₆Nb₆Si₇ and Fe can be expressed as (220)Fe₁₆Nb₆Si₇ // (110)Fe and the intergranular space were 0.38 nm and 0.17 nm, (020)Fe₁₆Nb₆Si₇ // (200)Fe and the intergranular space were 0.54 nm and 0.14 nm. Figure 5(i) evidences the highly coherent interface between the Fe₁₆Nb₆Si₇ and the Fe matrix. The FFT of the HR-TEM micrograph (figures 5(d), (e)) and the corresponding inverse fast Fourier transform (IFFT) pattern (figures 5(g), (h)) show parallel crystallographic axes of the Fe and Nb₇P₄ crystals: [130]Nb₇P₄ // [001]Fe, The orientation relationship of Nb₇P₄ and Fe can be expressed as (001)Nb₇P₄ // (110)Fe, and the intergranular space were 1.23 nm and 0.20 nm, (311)Nb₇P₄ // (110)Fe, and the intergranular space were 0.26 nm and 0.20 nm, figure 5(j) evidence the highly coherent interface between the and Nb₇P₄ the Fe matrix.

Figure 6(a) shows that Fe₁₆Nb₆Si₇ and Nb₇P₄ gather and grow together in a shape similar to ‘hoe’; The overlapped SAED patterns taken on the Fe / Fe₁₆Nb₆Si₇ interface (figure 6(b)) and the FFT of HR-TEM micrograph (figures 6(c), (d)) and the corresponding IFFT pattern (figures 6(f), (g)) show parallel crystallographic axes of these two crystals: [113]Fe₁₆Nb₆Si₇ // [112]Fe, The orientation relationship of Fe₁₆Nb₆Si₇ and Fe can be expressed as (220)Fe₁₆Nb₆Si₇ // (111)Fe, and the intergranular space were 0.40 nm and 0.20 nm, (242)Fe₁₆Nb₆Si₇ // (311)Fe, and the intergranular space were 0.18 nm and 0.09 nm, (422)Fe₁₆Nb₆Si₇ // (220)Fe, and the intergranular space were 0.23 nm and 0.11 nm. Figure 6(i) evidences the highly coherent interface between the Fe₁₆Nb₆Si₇ and the Fe matrix. The FFT of the HR-TEM micrograph (figures 6(d), (e)) and the corresponding inverse fast Fourier transform (IFFT) pattern (figures 6(g), (h)) show parallel crystallographic axes of the Fe and Nb₇P₄ crystals: [110]Nb₇P₄ // [112]Fe, figures 6(g), (h), (i) shows the incoherent interface between the and Nb₇P₄ the Fe matrix in these zone axes.

In summary, both Fe₁₆Nb₆Si₇ and Nb₇P₄ precipitates have a good coherent relationship with the Fe matrix. The precipitates with a good coherent relationship with the matrix have better grain boundary matching and lower interfacial energy, which is conducive to reducing the energy barrier in the nucleation process [18, 19], which is also the reason for a large number of precipitates.

Figure 7 shows the TEM images and EDS of particles after different annealing processes, the types of precipitates that were found by using the carbon extraction replica method. The number of precipitates was
large and the area density of 8.84 $\mu m^{-2}$ in annealed sheet subjected to 850 °C rolling (figure 7(a)) and the
dominant size of precipitates was in the range of 30–60 nm, the morphology of precipitates is mainly elliptical,
EDS spectrum showed that the components contained Fe, Nb, Si, S and Mn. EDS elemental mappings (figure 7(d)) evidence the precipitates were the diphase precipitates of Fe$_{16}$Nb$_6$Si$_7$ and MnS, where MnS is the
nucleation center and Fe$_{16}$Nb$_6$Si$_7$ grows up with it as the nucleation particle. No Nb$_7$P$_4$ precipitates were
founded showing that Nb$_7$P$_4$ precipitates have been dissolved in the steel during heat treatment at 850 °C.
The number of Precipitates was also large and the area density of 7.39 $\mu m^{-2}$ in annealed sheet subjected to
900 °C rolling (figure 7(b)) and the dominant size of precipitates was in the range of 40–70 nm, EDS spectrum
evidence the precipitates is Fe$_{16}$Nb$_6$Si$_7$.
The number of Precipitates decreased sharply and the area density of 4.66 $\mu m^{-2}$ in annealed sheet subjected
to 950 °C rolling (figure 7(c)) and the dominant size of precipitates was in the range of 40–80 nm, EDS spectrum
evidence the precipitates is NbC. When the heat treatment temperature was 950 °C and the holding time was
5 min, A large number of Fe$_{16}$Nb$_6$Si$_7$ precipitates were dissolved back to the steel, and the precipitates in the steel
were mainly NbC.
In order to better analyze the precipitation behavior of particles, Statistics of 30 transmission electron
microscope photographs. Determination of Volume Fraction of Second Phase Precipitates by Optimized
McCall-Boyd Method, The McCall-Boyd formula [20] is as follows:

$$V_f = \frac{1.46\pi}{6} \times \frac{N}{D^2} \times \frac{A}{A_s}$$

Where, $V_f$ = Volume fraction of particles, $N$ = Number of particles, $D$ = Average diameter of particles,
$A$ = Statistical area of particles.

The increase in yield strength ($\Delta\sigma_{ps}$) caused by precipitation strengthening can be estimated by the following
formula [21–24]:

$$\Delta\sigma_{ps} = 8.9952 \times 10^5 \times \frac{f^{1/2}}{d} \times \ln(2.417d)$$

Where, $f$ = Volume fraction of particles, $d$ = Average diameter of particles

The volume fraction of precipitates in the sample, the average particle diameter and the increment of
strength are shown in table 3. It can be seen that with the increase of heat treatment temperature, the average
diameter of precipitates increases, the volume fraction decreases, and the relative yield strength increment
decreases. The contribution of precipitates in the hot rolled sample to the strength improvement was maintained.
at about 100 MPa, the average diameter and total volume fraction of precipitates were 37.61 nm and 0.81%, respectively; After heat treatment at 850 °C, the average particle size of precipitates increases to 46.69 nm, and the volume fraction decreases to 0.76%, at this moment the increase of yield strength of steel by precipitates is about 80 MPa; After heat treatment at 900 °C, at this time, the average particle size of the precipitate is 53.39 nm, the volume fraction is 0.64%, and the increase in yield strength is about 66 MPa; After heat treatment at 950 °C, the average particle size of the precipitate is 61.46 nm, the volume fraction is 0.51%, and the increase in yield strength is about 52 MPa.

However, the second phase precipitation in the steel will distort the lattice, increase the dislocation density in the surrounding area, and further cause the internal stress field that is much larger than the volume of the precipitate itself, resulting in the change of the magnetic domain structure, the difficulty in moving the domain

Table 3. Precipitation relative intensity contribution.

| Heat treatment technology | Average diameter of particles / nm | Volume fraction of particles / % | $\Delta\sigma_p$ / MPa |
|---------------------------|-----------------------------------|---------------------------------|----------------------|
| Hot rolling               | 37.61                             | 0.81                            | 97.07                |
| 850 °C-5 min              | 46.69                             | 0.76                            | 79.38                |
| 900 °C-5 min              | 53.39                             | 0.64                            | 65.50                |
| 950 °C-5 min              | 61.46                             | 0.51                            | 52.27                |

Figure 7. TEM images and EDS of particles after different annealing processes. (a) 850 °C, 5 min; (b) 900 °C, 5 min; (c) 950 °C, 5 min; (d) EDS elemental mappings of the particle marked in figure 7(a).
wall and the difficulty in magnetization. Therefore, Hc (coercivity) and Ph (hysteresis loss) increase. The relationship between the size of precipitates and Hc (coercivity) is as follows:

\[ H_c \approx \frac{K}{\mu_0 M_s} \beta^{2/3} \times \frac{\delta}{d} \gg \delta \]

Where, \( \beta \) = Volume fraction of particles, \( d \) = Average diameter of particles, \( \delta \) = The thickness of magnetic domain wall, \( K \) = The anisotropic constants of magnetic crystals, \( \mu_0 \) = initial permeability, \( M_s \) = Saturated magnetization vector.

Formulas \( \delta, \mu_0 \) and \( M_s \) will change with the state of steel, which is difficult to measure. Therefore, it is impossible to calculate the specific value of the influence of the number and size of precipitates on the magnetic properties. However, it can be seen from the above equation that \( H_c \) is inversely proportional to the size of precipitates and is proportional to the number of precipitates. The greater the \( H_c \) value is, the greater the damage to the magnetic properties of non-oriented silicon steel is, which is contradictory to the increase of the relative yield strength of precipitates. Therefore, in actual production, it is necessary to adjust the size and quantity of precipitates according to the performance requirements of steel [25, 26].

4. Conclusion

In this study, two types of Laves phase of high Nb non-oriented silicon steel for new electric vehicles and their coherent relationship with Fe matrix were determined by SAED, FFT and IFFT, and calculation of steel strength enhancement by precipitation strengthening. The main conclusions are as follows:

1. The elliptical Laves phase is Fe\(_{16}\)Nb\(_6\)Si\(_7\) and the rectangular strip Laves phase is Nb\(_7\)P\(_4\) in the high Nb non-oriented silicon steel for the rotor of the drive motor of the electric automobiles. The orientation relationship between Fe\(_{16}\)Nb\(_6\)Si\(_7\) phase and Fe matrix is : [001]\(_{\text{Fe16Nb6Si7}}\) // [001]\(_{\text{Fe}}\) [101]\(_{\text{Fe16Nb6Si7}}\) // [110]\(_{\text{Fe}}\) [113]\(_{\text{Fe16Nb6Si7}}\) // [111]\(_{\text{Fe}}\) and the orientation relationship between Nb\(_7\)P\(_4\) phase and Fe matrix is : [130]\(_{\text{Nb7P4}}\) // [001]\(_{\text{Fe}}\) [102]\(_{\text{Nb7P4}}\) // [110]\(_{\text{Fe}}\) [110]\(_{\text{Nb7P4}}\) // [112]\(_{\text{Fe}}\).

2. Both Fe\(_{16}\)Nb\(_6\)Si\(_7\) and Nb\(_7\)P\(_4\) precipitates have a good coherent relationship with the Fe matrix.

3. The calculation results show that Fe\(_{16}\)Nb\(_6\)Si\(_7\), Nb\(_7\)P\(_4\) and other precipitates can improve the yield strength of non-oriented silicon steel, but at the same time, the magnetic properties of the steel will be damaged. Therefore, in actual production, the size and number of precipitates need to be appropriately adjusted according to the performance requirements of steel.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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