

## Fatigue Properties and Improvement Mechanisms of 1500 MPa-Class-High Strength Oil Tempered Wire

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Due to the increasing demand for carbon neutrality and resource saving, there is a demand for smaller and lighter spring products used in automobile engines, clutches, and other parts. As a result, it is necessary to increase the strength of spring materials. Conventionally, materials have been developed with the policy of increasing fatigue strength equating to increasing hardness. Spring materials, which require the highest fatigue strength among all metal materials, have reached a plateau, and a new approach that considers the environment in which the materials are used is becoming necessary. This paper describes the performance of a newly designed high-strength oil-tempered wire product.

Keywords: steel wire for springs, oil-tempered wire, fatigue strength, nitriding properties

#### 1. Introduction

Due to increasing environmental awareness, the technology required for automobiles is clearly changing from the conventional pursuit of high power and performance to the pursuit of lighter weight and higher fuel efficiency. However, the properties required for structural material used in automobiles have not changed: high strength (high tensile strength) and high fatigue strength. The theoretical strength of steel, the major structural material of automobiles, is very high and easily exceeds 10,000 MPa.(1) In view of the life cycle assessment, CO<sub>2</sub> emissions up to the manufacturing phase for steel are about one-fifth of those for aluminum and one-tenth of those for CFRP(2) due to its high recyclability, and the specific strength of high-strength oil-tempered wire\*1 is higher than that of Ti alloy, a light-weight, highstrength material. Therefore, the use of high-strength steel can be an effective solution to environmental issues.

High-strength springs are used for automobile transmissions, which especially require high strength. The thereby-obtained highly efficient power transmission and reduced weight greatly contribute to greater fuel efficiency.

Figure 1 shows the required properties of high-strength oil-tempered wire when it is used for automatic transmissions. Generally, four properties are required for high-strength springs: strength (tensile strength), fatigue strength, sag resistance,\*2 and toughness (defect sensitivity).

#### **Application example**

Damper spring for automatic transmissions

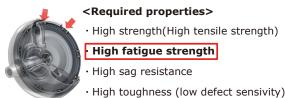


Fig. 1. Required properties of high-strength oil-tempered wire

Here, the highest level of fatigue strength of all types of metallic materials is required.

The development trend of high-strength oil-tempered wire by Sumitomo Electric Industries, Ltd. is summarized in Fig. 2. Since the successful commercialization of Chromium-silicon alloy steel oil-tempered spring wire for valve springs using our molten material in 1981, Sumitomo Electric has been promoting the development of material design technology. (3) In the mid-1990s, we developed VHS(1250 MPa-class), a high-Si, Cr-Si alloy steel oil-tempered wire, which has higher fatigue strength. (4) During the development, we broke away from the conventional idea that high-strength material leads to springs with high fatigue strength. There were two development concepts: one was higher strength and toughness for the springs, and the other was higher fatigue strength with finer grains.

In the 2000s, we developed VHR(1350 MPa-class), a high-Si, high-Cr oil-tempered spring wire that has a higher fatigue strength than VHS.<sup>(5)</sup> To take the development concept of VHS even further, we carried out the development with the customers' spring manufacturing process in mind. Before developing VHR, we had set the target of

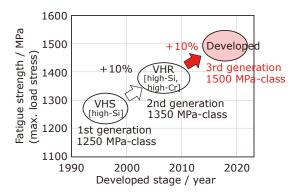


Fig. 2. Trend of oil-tempered wire for high-strength springs in Sumitomo Electric

promoting nitriding on the surface of springs during the nitriding process, which determines the properties of high-strength springs, without deteriorating the internal hardness, and achieving strength and toughness equivalent to those of current high-strength steel.

In the development of the third-generation new steel, we set the target of 1,500 MPa-class, high-strength oil-tempered wire for springs, improving fatigue strength by an additional 10% in comparison with VHR (hereinafter referred to as the "current high-strength material") to produce the highest-strength material that can properly respond to customer demand even in the market for power-trains of internal-combustion engines, which is expected to shrink from now on.

#### 2. Product Development Concept

The general manufacturing process of compression coil springs is shown in Fig. 3. After the coiling process, annealing to relieve excessive stress is applied. Then, nitriding treatment\*3 to harden the surface and shot peening treatment\*4 to apply compressive residual stress and smooth the spring surface are applied. Finally, additional annealing is applied. The abovementioned 1,500 MPa-class fatigue strength of springs must be obtained after the process of these working and heat treatments, making the material design very difficult.

Figure 4 shows the internal stress state in the near-surface inside of a spring, which produces the maximum stress when the springs are working. Under practical-use conditions, fatigue fractures are usually observed near the surface of a spring because the stress borne by a spring is the highest on the spring's surface. Therefore, the following treatments are adopted for the manufacturing process of springs, especially high-strength springs: nitriding treatment to harden the surface and shot peening treatment to apply compressive residual stress on the surface to lower the actual stress (Fig. 3). However, the heat effects of the nitriding treatment cause the internal hardness of the spring to decrease, resulting in internal fatigue fractures (Fig. 4). The other problem is that strengthening steel wire deteriorates the wire's toughness and causes fatigue fracture from inclusion.\*5 Therefore, when developing the new steel, we set the following three targets: (1) improving nitriding performance (surface hardness), (2) improving heat resistance (yield strength), and (3) maintaining high toughness.

Table 1 describes the factors in spring fatigue strength and measures for improvement in consideration of these factors. In the table, 1 shows factors in strength and 2 shows a factor in toughness. The effects of the factors in strength ① vary considerably, mainly depending on the chemical composition of the steel, but the spring elastic limit can be improved by adjusting the conditions of the continuous heat treatment process (oil-tempered process, hereinafter referred to as the "OT process"). In addition, the effects of the factor in toughness ② also depend greatly on the composition, but it can also be further improved by the OT process conditions. In other words, we aimed to improve the spring fatigue strength not only by using a conventional method of composition design but also by establishing a heat treatment method suitable for the chemical composition of the newly developed material.

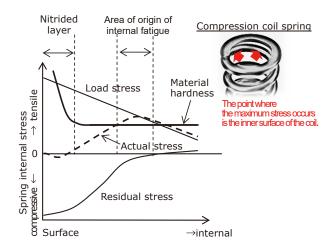


Fig. 4. Internal stress state of spring (near-surface inside of spring)

Table 1. Factors in spring fatigue strength and measures

	Factors in spring fatigue strength	Improvement measures	Approach	
1	Nitriding performance (Surface strength)	Composition	Conventional measures * However,	
	Internal strength after nitriding	Composition	considering the spring manufacturing process	
	Spring elastic limit (0.2% proof stress)	Heat treatment	Newly developed measures	
2	toughness (Inclusion sensitivity)	Composition, Heat treatment	Newly developed measures	

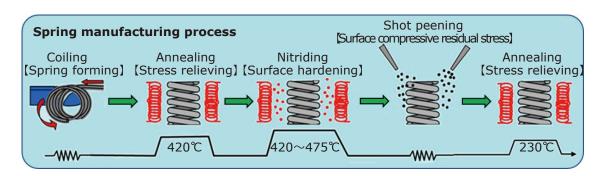


Fig. 3. General manufacturing process of compression coil springs

#### 3. Material Design and Manufacturing Condition Establishment

#### 3-1 Material design

The chemical compositions of the developed material, the current high-strength material, and the Cr-Si alloy steel general-purpose material (JIS SWOSC-V, SAE 9254) for reference are shown in Table 2. We conducted trial mass production in collaboration with a steel manufacturer, and obtained sample wire through the same process as the current high-strength steel process. In the table, the composition of SWOSC-V shows the values described in JIS.

Table 2. Chemical composition of new steel from trial mass production [mass%]

Туре	С	Si	Mn	Cr	V	Others
Developed material	0.65	1.86	0.37	1.77	0.19	_
Current High-strength material	0.65	2.21	0.55	1.17	0.14	Co:0.2
Ref.) SWOSC-V(JIS)	0.51 ~0.59	1.20 ~1.60	0.50 ~0.80	0.50 ~0.80	_	_

As described above, when developing the new steel, we aimed to improve the properties of springs made of the new material, and to establish the optimal chemical composition and manufacturing conditions.

As for the chemical composition, the same fundamental design concept as for current high-strength steel was applied. The chemical composition was increased in Si, Cr, and V, strengthening the components of Cr-Si alloy steel as a basis for the new steel.

Si, an element of the composition, dissolves in ferrite and increases softening resistance at high temperatures, preventing the decrease in material strength during the abovementioned heat treatment in spring manufacturing. This results in (1) higher tempering temperature in the OT process, which enables reduced microscopic defects in the metallographic structure and improves toughness, resulting in higher spring machinability; (2) higher annealing temperature for stress relief in the spring manufacture, which enables the removal of excessive stress developed during the coiling process, resulting in higher sag resistance, and reduces microscopic defects (a cause fatigue fractures) in the metallographic structure, improving fatigue strength; and (3) higher temperature of the nitriding treatment in spring manufacture, this makes nitrogen diffusion easy, causes the nitriding to penetrate deeper, and minimizes the decrease in hardness of the deeper interior that is unnitrided and heat-affected, resulting in higher fatigue strength. However, since Si has the property of being easily combined with oxygen, excessive addition leads to the concentration of Si on the spring surface due to its oxidizability, and tends to inhibit the inward diffusion of nitrogen. Additionally, since Si is a component element of nonmetallic inclusions, the amount of Si was slightly reduced in comparison to the current high-strength steel.

Secondly, the reason the amount of Cr was increased is shown below. Cr is a representative carbide former, and its high affinity with carbon promotes carbide refining, (6),(7) improving softening resistance. Cr is also a component element of nitride and helps nitrogen penetration during the

nitriding treatment, promoting hardening. Dispersion precipitation of nitride produces compressive residual stress around the surface, greatly improving fatigue strength.

Thirdly, the reason the amount of V was increased is explained below. V is also a representative carbide former and helps form fine carbide that is relatively stable under high temperature in steel, resulting in finer prior  $\gamma$  grains and higher softening resistance. In addition, it helps form nitride in the lattice of ferrite in the nitriding treatment and suppresses slip caused by repeated stress, contributing to higher fatigue strength.

As with Si, Mn is added as a deoxidizer. However, the effect is limited in the case of newly developed material to which a large amount of Si is added, and Mn tends to generate bainite, martensite, and other supercooling structures during hot rolling and patenting treatments, lowering drawing machinability. Therefore, the amount of Mn was reduced in comparison to current high-strength steel.

As described above, in this development, we did not use the conventional method of increasing the hardness and strength of the material by adding or increasing the amount of steel-strengthening elements, but rather focused on improving the spring characteristics.

#### 3-2 Manufacturing condition establishment

The final manufacturing process of oil-tempered wire is the OT process, and optimizing the heat treatment conditions in the process determines the properties of the oil-tempered wire products. Figure 5 shows the hypothesis for optimizing the heat treatment conditions. The previous chapter describes the factors in spring fatigue strength and measures. The new heat treatments require improvement of the elastic limit and toughness of springs. There are three methods of improving the elastic limit. One is producing finer grains (based on the Hall-Petch relationship), another is reducing insoluble carbide, and the other is fine carbide dispersion (the Orowan mechanism). There are two methods of improving toughness. One is reducing insoluble carbide, and the other is fine carbide dispersion. To improve the required properties, we used the common denominator: heat treatment at a higher temperature for a shorter time in the OT process, as shown in the figure.

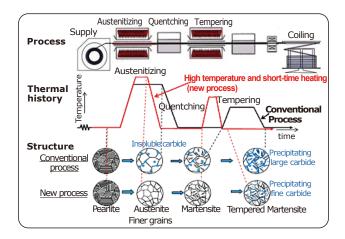


Fig. 5. Heat treatment hypothesis to improve the spring fatigue strength of developed material

In this development, we refined the new material and established the high-temperature short-time heat treatment by determining the new OT process conditions, such as the heating temperature and the holding time, designed specifically for the developed material. The prior  $\gamma$  grain size of the developed material is shown using the grain size number\*6 in Table 3. Since the developed material includes more elements composing carbide and uses higher temperature to reduce insoluble carbide, it is prone to enlarged grain sizes. By minimizing the heating time, the grain size growth is suppressed and a finer structure is achieved.

Table 3. Prior γ grain size of developed material

Туре	Grain size Number		
Developed material	12.5		
Current high-strength material	12.4		

#### 4. Results

#### 4-1 Tensile properties

The tensile properties of the developed material (n = 3) are shown in Table 4. As a reference, the JIS standard for general-purpose Cr-Si alloy steel materials is also shown.

This time, we used a trial product with a diameter of 3.5 mm for evaluation. The tensile strength of the developed material was adjusted to about 2,150 N/mm² in consideration of spring machinability. The tensile test revealed that the reduction of area was smaller than that of the current high-strength material but was 45% or more, showing high toughness.

Table 4. Tensile properties of developed material

Туре	Diameter (mm)	Tensile strength (N/mm²)	Reduction of area (%)	Elongation (%)
Developed Material	3.50	2158	45.6	5.2
Current high- strength material	3.50	2153	49.5	6.0
JIS G3561 Grade SWOSC-V	3.50	1860~2010	≥ 45	_

# 4-2 Temper properties (annealing-softening resistance)

The tensile test results of the developed and current highstrength materials (n = 3) after low-temperature annealing at temperatures of 400–475°C for 30 minutes in the atmosphere, assuming stress relief annealing after spring machining, are shown in Fig. 6. In the figure, tensile strengths are shown as lines and reductions of area are shown as bars on the secondary axis. After annealing, the tensile strength of the developed material becomes highest at a temperature of 425°C. In comparison with the current high-strength material, the tensile strength of the developed material is lower up to a temperature of 425°C and is equal at a temperature of 450°C. At a temperature of 475°C, the developed material has a higher tensile strength and softening resistance. This shows that Si contributes to higher softening resistance at relatively low temperatures, while Cr and V contribute to the same at high temperatures. The reduction of area of the developed material is highest at a temperature of 400°C and shows little difference at temperatures of 400–475°C, showing stable and high toughness at high temperatures.

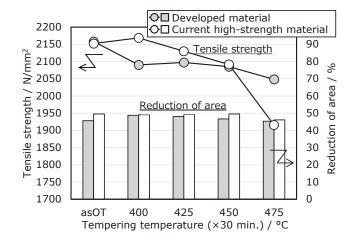


Fig. 6. Temper properties of developed material (annealing-softening resistance)

#### 4-3 Sag resistance

Then U-shaped tests were conducted to evaluate sag resistance. Figure 7 shows an overview of the test.

In a U-shaped test, a wire is bent into a U shape. One end is fixed and the other end is twisted to a certain angle to apply a shearing stress, assuming the actual use of the wire as a spring. It is held in a heated furnace for a certain period of time. After the test, the sample is taken out of the furnace. After the shearing stress is released, the residual twist angle is measured to evaluate the residual shearing strain. Low residual shearing strain means high sag resistance. This time, we adopted a test method that assumes a harsh usage environment for general damper springs. After being bent into a U shape, samples were heated at a temperature of 430°C for 3.5 hours (equivalent to nitriding treatment), subjected to a shearing stress of 1,200 MPa at a room temperature, and held in a furnace at a temperature of  $165^{\circ}$ C for 24 hours. The results (n = 3) are shown in Table 5.

The developed material was confirmed to have higher sag resistance than current high-strength materials.

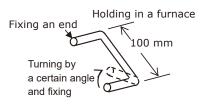


Fig. 7. Overview of U-shaped test method

Table 5. Sag resistance of developed material

Туре	Diameter (mm)	Thermal treatment	Applied Load stress (MPa)	Residual shear strain (%)
Developed material	3.50	430°C × 3.5 hrs.	1200	0.252
Current high- strength material	3.50	"	"	0.287

#### 4-4 Fatigue properties

To evaluate the fatigue strength of the developed material, Nakamura-type rotating-bending fatigue tests\*7 were conducted. To estimate the fatigue properties of springs, the samples were tempered at a temperature of 420°C for 30 minutes. In consideration of the decrease in internal hardness by the heat effects of nitriding, the samples were heated at a temperature of 430°C for 3.5 hours and the fatigue tests were conducted. The obtained S-N curves are shown in Fig. 8.

In comparison with the current high-strength material with a fatigue strength of 1,030 MPa, the developed steel has a fatigue strength of 1,100 MPa, showing an improvement of about 7%.

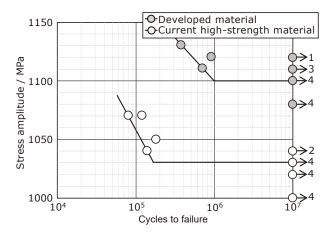


Fig. 8. Fatigue properties of the developed material

#### 4-5 Nitriding properties

To evaluate the nitriding properties of the developed material, after stress relief annealing at 420°C for 30 minutes, oxide films on the surfaces of samples (added to improve spring machinability) were removed by shot blasting treatment. Nitriding treatment was applied at a temperature of 430°C for 3.5 hours.

First, the hardness distribution around the surface before nitriding (n=4) is shown in Fig. 9. This shows that the hardness of both the developed and the current high-strength materials is decreased from the very surface to a depth of around 0.02 mm due to decarburization. Before nitriding treatment, there is little difference in hardness between the developed and current high-strength materials, as is the case with tensile strength, and the current high-strength material is slightly harder than the developed material.

Next, the hardness distribution around the surface

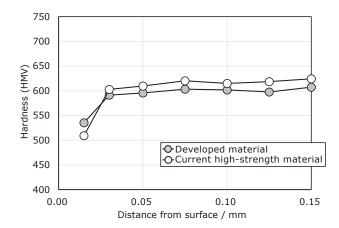


Fig. 9. Hardness distribution around the surface of the developed wire and the current high-strength wire (as OT, before nitriding)

after nitriding (n = 4) is shown in Fig. 10.

This shows that the developed material has high hardness, especially in the vicinity of the surface. Considering that the Vickers hardness of both the developed and the current high-strength materials is about 600HV0.2 at a depth of 0.10 mm or more due to the heat effects during nitriding, the nitriding treatment hardened the developed material more around the surface than the current high-strength material, and hardened deeper areas of the developed material.

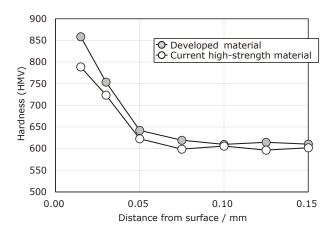


Fig. 10. Hardness distribution around the surface of the developed material and the current high-strength material after nitriding  $(430^{\circ}\text{C} \times 3.5 \text{ h})$ 

#### 5. Conclusion

The results of these development activities are summarized as follows:

- We have developed the highest-strength 1,500 MPa-class oil-tempered wire, especially with the customers' spring manufacturing process in mind.
- When developing the new steel, we set three targets: (1) improving nitriding performance (surface hardness), (2) improving heat resistance (yield strength), and (3) maintaining high toughness.

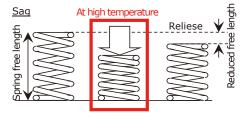
- We aimed to improve spring fatigue strength not only by using a conventional method of composition design, but also by establishing manufacturing conditions suitable for the chemical composition of the newly developed material.
- We found that the developed material has high levels of both strength and toughness after evaluation. The softening resistance after heat treatment, such as annealing, is higher than that of the current highstrength material.
- The sag resistance evaluation revealed that the developed material has higher properties than the current high-strength material.
- We conducted fatigue tests in consideration of the heat effects of nitriding (decreasing internal hardness), and found that the developed steel has higher fatigue strength than the current high-strength material by about 7%.
- After the nitriding property evaluation, we confirmed that the nitriding treatment further increased the hardness of the developed material and promoted nitriding to a deeper area.

From these evaluation results, we expect that springs made of the developed material will show much higher fatigue strength in an evaluation than those made of the current high-strength material.

We have already supplied the developed material to some customers for evaluation, and their expectations of 1,500 MPa-class, next-generation, high-strength material are being satisfied. We will further improve the material to suit various applications and to meet widespread market expectations.

#### **Technical Terms**

- \*1 Oil-tempered wire: Heat-treated steel wire that has a tempered martensite structure produced by a quenching-tempering continuous heat-treatment process. It has the features of high (fatigue) strength and high heat resistance.
- \*2 Sag resistance: Resistance to sag, which is a phenomenon that occurs when a spring used for automobile parts is working at high temperatures of over 100°C under load stress, resulting in the reduction of its free length and loss of its original restoring force.



- \*3 Nitriding treatment: A process that hardens the surface of steel to improve fatigue strength and wear resistance. In nitriding treatment, steel is heated to around 400–500°C in ammonia gas, for example, to precipitate carbonitride in the steel, and a very hard surface is obtained. It is a common method to manufacture high-strength springs made of oil-tempered wire, and improves fatigue strength significantly.
- \*4 Shot peening: A spring manufacturing process of hitting the surface of a spring at high speed with a large number of small balls made of steel or nonferrous metal or steel wire cut to lengths approximately the same size as the diameter of the spring wire. This process causes plastic deformation that results in surface smoothing, strain hardening, and compressive residual stress, as well as higher fatigue strength.
- \*5 Inclusion: During steelmaking, deoxidizers are used to remove oxygen from steel. A portion of the deoxidizing agent that has reacted with oxygen to form oxides cannot be completely removed, and what remains is nonmetallic inclusions. We lower the melting point by adjusting the composition, etc., and try to make it harmless in the rolling process.
- \*6 Prior austenite (γ) grain size, grain size number: An index of grain size, specified as follows (according to JIS G 0551):

 $b=2^{(n\,+\,3)}$ 

where n is the grain size number, and b is the number of grains per 1mm2 of the material cross section. Recently, grain-size refinement has become a keyword in material development. Discussions on oil-tempered wire are often focused on the grain sizes (prior  $\gamma$  grain size) of austenitized structures while being heated during the quenching process. The prior  $\gamma$  grain size is measured by microstructural observation.

Nakamura-type rotating-bending fatigue test: A fatigue test involving the rotation of a steel wire sample to which a constant bending moment is applied by four-point bending, and the application of repeated bending stress on the steel wire surface at a stress ratio of -1. In a general steel wire evaluation for springs, fatigue strength (fatigue limit) is defined as the maximum stress after  $1 \times 10^7$  cycles of stress under the elastic limit without fatigue failure. Actual springs are used after low-temperature annealing or nitriding treatment. Therefore, to identify the properties under conditions as close as possible to the actual conditions, the fatigue tests for steel wire are conducted after heat treatment (at 420°C for 20 minutes), which is intended to represent low-temperature annealing after coiling, heat treatment (at 430°C for 3.5 hours), which is intended to represent nitriding, and selective shot peening to steel wire for the purpose of eliminating the effects of the rough surface.

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