High-toughness Heavy Section Steel Plate with Minimum Yield Strength of 900 MPa for Deep Submergible Vehicle

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Synopsis
Steel intended for use as the hull materials of deep submergible vehicles has been developed. The target properties of the steel were minimum yield strength of 900 MPa and high toughness.
The primary research was conducted on several steels containing a range from 2.5 to 5% Ni with the emphasis on mechanical properties, heat treating characteristics and weldability. The optimum chemical composition of steel having the above-mentioned properties has been formed to be 0.09C−5Ni−0.5Cr−0.5Mo−0.05V with minimal impurities, especially, low sulfur content. Production heats of this steel were conducted. Slab was made by electroslag remelting process for having ultra low impurities and super cleanliness. Plates of quenched and tempered steel of 65 mm in thickness exhibited yield strength having a range from 931 to 960 MPa, and Charpy V-notch shelf energy absorption having a range from 172 to 209 J.
According to this good quality, this steel plate was applied for the hull material of deep submergible vehicle (“Shinkai 2000”) operating in seas of 2,000 m depth.

I. Introduction
In recent years, offshore development in general and scientific exploration of the seabed in particular have been attracting attention in parallel with space exploration. As oceanic exploration is directed toward greater depths, submersibles must be capable of descending to greater depths and their pressure hull must be strong enough to withstand the enormous pressures encountered. To this end, it is necessary to increase the strength of the pressure hull material, decrease its section thickness and raise its strength-to-weight ratio.
The deep submersible has a spherical pressure hull.
If the pressure hull is made of steel, the higher the yield strength of the steel, the deeper the hull can descend.
If the pressure hull is made of steel with a yield strength of the 900-MPa class, for example, the hull can theoretically descend to a depth of about 4,000 m and explore up to about 50% of the world’s ocean floors.
Japan has developed steels for deep-sea submersibles with yield strength of about 450 and 620 MPa.
Elsewhere, chiefly in U.S.A., HY80 with a yield strength of the 550-MPa (80-ksi) class was commercially applied, followed by HY130, 18% Ni managing steel with a yield strength of the 1,240-MPa class, and 10Ni−Cr−Mo steel.
Against this backdrop, the authors have developed an extra-heavy-gauge, high-strength and high-toughness steel plate through the application of the electroslag remelting (ESR) process to obtain improved cleanliness and homogeneity. The new steel contains relatively low amounts of alloying elements and has a yield strength of 900 MPa.

II. Properties Required for Development Target
A yield strength of 900 MPa, 280 MPa higher than that of conventional steel plate for submersibles, was established as the target strength.
Minimum toughness requirements established are Charpy V-notch impact energy (\(\gamma E_{\text{Charpy}}\)) of 70 J as based on the works of Pellini et al., and Charpy V-notch impact energy (\(\gamma E_{\text{Charpy}}\)) of 70 J at 190 K in consideration of nil-ductility transition (NDT) temperature 190 K in drop-weight tests. These toughness requirements must also be met in the heat-affected zones of welds. In addition, the pressure hull steel plate must have good corrosion resistance and fatigue properties in seawater.

In general, high-strength steels are produced by using a nickel-bearing steel as the basic composition steel and creating a fine tempered-martensitic structure through grain refinement. The steel that is the subject of this report is also based on this concept. The pressure hull of a deep-sea submersible is spherical in shape. After fabrication into the sphere, the extra-heavy-gauge steel plate must be heat treated (quenched and tempered). This calls for a steel with extremely stable heat treatment characteristics, such as hardenability. The pressure hull is then ground to make it a true sphere. The steel must be so thick and clean that this grinding operation does not reveal any segregation, nonmetallic inclusions or internal defects.

III. Experimental Procedures
1. Study of Basic Composition Steel
The test steels have chemical composition of range from 0.05 to 0.11% C, 2.5 to 5% Ni, 0.4 to 0.8% Cr, 0.3 to 0.6% Mo and 0 to 0.06% V. The chemical compositions of four representative test steels are

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given in Table 1. They are a 2.5Ni-Cr-Mo steel; a 3Ni-Cr-Mo-V steel which has a slightly higher nickel content and utilizes the precipitation hardening of vanadium plus molybdenum; a 4Ni-Cr-Mo-V steel which has a lower carbon content and a higher nickel content and uses the precipitation hardening of molybdenum and vanadium for strengthening; and a 5Ni-Cr-Mo-V steel with a still higher nickel content.*

Each test steel was manufactured as follows: 100-kg vacuum melting and 1-t electric arc furnace melting → teeming into 100 to 250-kg ingots → reheating at 1 470 K and plate rolling → plate finishing at 1 220 K (plate thickness of 50 to 65 mm) → air cooling. The test steels thus obtained were investigated for the effects of heat treating conditions on mechanical properties and for the toughness of welding heat-affected zones, as described below.

1. Effects of tempering temperature on strength and toughness of each steel. In this case, the quenching temperature was 1 110 K and the tempering temperature was 870 to 930 K.

2. Effects of quenching and tempering conditions on strength and toughness of optimum steel. Two quenching treatments (Q1 and Q2) were given to simulate actual practice. The reason is as follows: When fabricating the pressure hull of a deep-sea submersible, the steel plate is hot worked into a sphere. The fabricated pressure hull is first given the Q1 heat treatment to remove the effects of hot working and is then given the Q2 heat treatment to impart the desired properties. The Q1 and Q2 heat treatments were performed at temperatures of 1 120 to 1 200 K and 1 050 to 1 120 K, respectively, for a time of 60 min each in consideration of the A1 transformation point. The effects of cooling rates of Q1 and Q2 quenching were examined over a range of 1.1 to 3.9 K/sec. The cooling rate was controlled by fog cooling (1.1 to 3.2 K/sec) and by direct water quenching (3.9 K/sec). With reference to the results of Paragraph (1) above, the tempering temperature was set at 870 K and time at temperature was varied over a range of 60 to 180 min.

3. Toughness of welding heat-affected zone. A welding heat cycle simulation test was conducted on the steel, which had optimum heat treatment characteristics, to investigate the toughness of heat-affected zones. The steel was heated to 1 620 K, cooled at various rates and inspected for the toughness of the heat-affected zone.

2. Commercialization Tests

According to the results of study on the basic composition steels with the use of steels melted on a small scale, manufacturing tests by large-scale melting were then carried out on the optimum composition steel (5 Ni steel as described later). The steel was melted in a 60-t electric arc furnace and cast into ingots weighing 16 to 18 t. Since the steel must have minimum segregation, be highly homogeneous and be almost free of internal defects, the ingot was first rolled into a slab of desired size and the ESR process was then applied with the slab serving as the electrode. The electroslag-remelted slab was then rolled into a plate of 65-mm thickness and heat treated under the optimum conditions mentioned above.

The steel plates thus manufactured were tested for properties as described below.

JIS No. 4 tensile test specimens were tensile tested for yield strength (YS), tensile strength (TS), elongation (EL) and reduction of area (RA). JIS No. 4 impact test specimens were Charpy V-notch impact tested at temperatures of 100 to 290 K for Charpy V-notch impact energy (VE). As large fracture tests, the steel was also dynamic tear (DT) tested according to ASTM procedures in order to determine its brittle fracture transition temperature.

Various heat treated specimens and welding heat cycle simulation tested specimens were observed for optical, extraction replica and thin-foil transmission electron microstructures and determined for the precipitated austenite phase by X-ray diffraction, as required. Continuous cooling transformation (CCT) diagrams according to thermal simulator were developed for some of the test steels.

IV. Experimental Results

1. Selection of Basic Composition Steel

The effects of tempering temperature on the yield strength and toughness of each test steel are shown in Fig. 1.

Tempering the 2.5 Ni steel at temperature of 870 to 900 K imparts a yield strength of about 700 to 750 MPa, far short of the target value of 900 MPa. Relatively high in carbon, the 3 Ni steel has a higher yield strength, but its yield strength greatly varies

<table>
<thead>
<tr>
<th>Steel type</th>
<th>Thickness (mm)</th>
<th>Chemical composition (wt%)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
<td>Si</td>
</tr>
<tr>
<td>2.5Ni</td>
<td>50</td>
<td>0.10</td>
</tr>
<tr>
<td>3Ni</td>
<td>60</td>
<td>0.11</td>
</tr>
<tr>
<td>4Ni</td>
<td>65</td>
<td>0.06</td>
</tr>
<tr>
<td>5Ni</td>
<td>65</td>
<td>0.09</td>
</tr>
</tbody>
</table>

* For the sake of brevity, the 2.5Ni-Cr-Mo steel may be sometimes written as 2.5 Ni steel, 3Ni-Cr-Mo-V steel as 3 Ni steel, 4Ni-Cr-Mo-V steel as 4 Ni steel and 5Ni-Cr-Mo-V steel as 5 Ni steel.
with the tempering temperature and the optimum tempering temperature range is narrow. Therefore, yield strength stability will become a problem in commercial applications. The 4 Ni steel with a low carbon content can obtain a yield strength of 920 MPa at a tempering temperature of, for example, 870 K, but has no margin for the target yield strength of 900 MPa. In contrast, the 5 Ni steel can stably develop a yield strength of 900 to 1 000 MPa at tempering temperatures of 870 to 910 K. This steel also combines high yield strength with high toughness.

Carbon is one of the elements that most markedly affect the yield strength of steel. Figure 2 illustrates the effect of carbon on yield strength with the 5 Ni steel taken as the basic composition steel. According to this figure, the optimum carbon content is not less than 0.07 % or ranges from 0.08 to 0.09 %. As a result, the 5 Ni steel with a carbon content of 0.08 to 0.09 % was selected as the basic composition steel which is capable of developing a yield strength of not less than 900 MPa when quenched and tempered.

2. Heat Treatment Characteristics of 5 Ni Steel

1. Continuous Cooling Transformation (CCT) Diagram

The CCT diagram which shows the basis for heat treatment characteristics is given in Fig. 3. The Ac1 point is 944 K and the Ac3 point is 1 024 K. High-nickel steels, however, have true and apparent Ac1 points. The figure presents the latter point. When the 5 Ni steel is cooled over a considerably wide cooling rate range from 1 160 K, a structure of martensite or martensite and some lower bainite is obtained. This means that the 5 Ni steel has good hardenability.

2. Effects of Quenching Conditions

The effects of quenching temperature on strength and toughness are presented in Fig. 4. The yield strength tends to increase with increasing quenching temperature. Raising the Q1 and Q2 temperatures from 1 120 to 1 200 K and from 1 050 to 1 120 K, respectively, increases the yield strength by 35 to 40 MPa. The tensile strength exhibits an almost identical tendency. The ductility and toughness are hardly affected by the quenching temperature and show good results under each set of the quenching conditions studied.

Photograph 1 shows an optical microstructure (as quenched) when the quenching temperature was changed. As the quenching temperature rises, the austenite grain diameter increases and along with this growth, slightly coarsened acicular martensite appears.

Figure 5 shows the effects of cooling rate of quenching on strength and toughness when the Q1 and Q2 temperatures were 1 120 and 1 050 K, respectively. The cooling rate exerts a pronounced effect on the yield strength. To satisfy the target yield strength of 900 MPa, the cooling rate must be at least 2 K/
When water quenching is employed, a cooling rate of over 3 K/sec is obtained. At this cooling rate, the yield strength of 900 MPa can be satisfactorily obtained. The cooling rate similarly affects the tensile strength, but not so conspicuously.

3. Effects of Tempering Conditions

As discussed in Section IV.1, the 5 Ni steel can stably develop a minimum yield strength of 900 MPa at a tempering temperature of 870 to 910 K. The effects of time at the tempering temperature of 870 K on the strength and toughness of the steel were investigated. The results are plotted in Fig. 6. As can be clearly seen from the figure, both strength and toughness are affected little by the time at the tempering temperature.

To investigate the cause for the stably good strength and toughness of the 5 Ni steel, the change in optical microstructure with tempering temperature was observed. The results are shown in Photo. 2.

The 5 Ni steel exhibits a fine-grained and optimum low-carbon tempered-martensitic structure at the tempering temperature of 870 to 900 K. As the tempering temperature rises to 930 K, tempered-martensitic structure coarsen. This agrees well with the phenomenon that the yield strength substantially decreases at the tempering temperature of 930 K as previously discussed in the section on the yield strength variation with tempering temperature.
Extraction replica and thin-foil transmission electron microstructures at the tempering temperature of 900 K are given in Photo. 3. The extraction replica microstructure shows carbides finely dispersed and precipitated in tempered martensite, while the transmission electron microstructure shows carbides finely dispersed and precipitated in acicular or massive tempered martensite.

Austenite precipitation by tempering is reported to be generally effective in improving the toughness of high-nickel steels. As follow-up experiment, the amounts of the austenite phase precipitated in the 5 Ni steel and in the 4 Ni steel for comparison at the tempering temperatures of 870, 890, and 900 K were determined. The results are given in Table 2. The determined amount of precipitated austenite is 3 to 4 % at the tempering temperatures of 870 to 900 K. About 1 % was confirmed to be precipitated in the 4 Ni steel at the tempering temperature of 900 K.

3. Toughness of Heat-affected Zone of 5 Ni Steel in Welding Heat Cycle Simulation

Figure 7 shows the toughness change of the 5 Ni steel when it was cooled for different times from the maximum heating temperature of 1 620 K in welding heat cycle simulation tests. As the cooling time extends, the toughness tends to decrease, but the degree of decrease is small. Over a wide cooling time range,$E_{190}$ is at least 100 J, indicating good toughness.

4. Commercial Production Test of 5 Ni Steel

The 5 Ni steel (0.09C-0.6Cr-0.5Mo-0.05V steel) judged optimum for the target properties as described above was produced on a large commercial scale with impurities, particularly sulfur, suppressed, and was investigated for resultant properties.

The chemical composition analyses of the slab produced by the ESR process are shown in Table 3. The electroslag remelted slab was hot rolled to a 65-mm thick plate and heat treated. The $Q_1$ and $Q_2$ quenching temperatures were 1 120 and 1 050 K, respectively, and the tempering temperature was 870 K. The mechanical properties of the heat-treated plate are given in Table 4. The plate has good properties in the through-thickness direction as well and has a minimum of anisotropy, satisfying the target strength and toughness requirements. The transition
temperature confirmed in the dynamic tear (DT) test from the standpoint of fracture toughness was 180 K. In addition, good results are obtained with respect to weldability, corrosion resistance and fatigue properties.16,17)

V. Discussion

High-strength steels are generally produced by utilizing the fine tempered martensite developed through quenching and tempering. The 5 Ni steel is also based on this structure and takes advantage of precipitation hardening in addition (Photo. 3). Because of its extraheavy gauge of 65 mm, the steel contains a large amount of nickel to improve its hardenability (Fig. 3).

Besides the fine tempered-martensite matrix of the nickel steel, the precipitation of fine austenite is a noteworthy factor for increased toughness. The dispersion and precipitation of fine austenite in the ferrite matrix is believed to act most favorably for the prevention of fracture initiation and propagation. Lessening the amounts of impurities and inclusions is also effective in increasing toughness. To improve $\nu_{\mathrm{y}}$, sulfur as well as phosphorus must be decreased.6) In this connection, the application of the ESR process proved extremely effective in increasing toughness. Sulfur content reduction is also effective in lowering welding crack susceptibility,18,19) Since the pressure hull of a deep-sea submersible must meet very close tolerances, the steel plates fabricated into the sphere are ground. This grinding operation must not reveal any nonmetallic inclusions or internal defects. For this reason, the steel plate must be extremely clean and must be virtually free of internal defects. In this respect, the application of the ESR process to this steel has been a major technological point for the successful commercial production of the steel.

VI. Summary

To develop a high-strength and high-toughness steel plate of the 900-MPa yield strength class for the pressure hull of deep-sea submersibles, steels with 2.5 to 5.0 % Ni were investigated for heat-treatment characteristics and resultant mechanical properties. As a result, a 5 Ni steel (0.09C–Ni-0.6Cr-0.5Mo-0.05V steel) was selected as the optimum composition steel and was subjected to confirmatory tests on a commercial scale by the use of the electroslag remelting process.

Optimum heat-treating conditions were double quenching and tempering. The first quenching temperature was 1 120 to 1 200 K, the second quenching temperature was 1 050 to 1 120 K, and the cooling rate was 2 K/sec or higher in each stage. The tempering temperature was 870 to 900 K. The 65-mm thick steel plates produced under these conditions were of extremely high quality, with a yield strength of over 900 MPa, a minimum of anisotropy and a transition temperature of 180 K under dynamic tear (DT) test to determine fracture toughness.

Acknowledgements

The authors gratefully acknowledge the cooperation of personnel concerned at the Kobe Shipyard of Mitsubishi Heavy Industries, Ltd. in this study.

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