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The body can be viewed from the next page.

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1 Introduction

In recent years, the drilling for oil and natural gas has been made at greater depth where the environment becomes aggravated by the presence of CO_2, H_2S, Cl^-, etc. Because conventional carbon steel or low alloy steel tubes entail corrosion problems when used in these wells, a new demand is increasing for 13 Cr stainless steel, duplex stainless steel, and high nickel alloys as OCTG materials.

In order to comply with these worldwide needs, Chita Works of Kawasaki Steel has established a manufacturing system for upset tubings of 13 Cr stainless steel by developing, since 1982, rolling techniques on the mandrel mill, upsetting, and heat treatment of such stainless steels.

This paper describes manufacturing techniques and characteristics of 13 Cr stainless steel tubings based on the manufacturing results of premium joint tubing of 2-7/8″ × 6.5 lb/ft (API 5ACL-80).

2 Needs for Developing 13 Cr Stainless Steel and its Property Requirements

The corrosive environment of deeper oil or gas wells now under exploratory drilling becomes increasingly severe with an increased concentration of CO_2, H_2S, and Cl^-. In addition, the following problems have limited the use of inhibitors and promoted the increased application of 9 Cr steel, 13 Cr stainless steel, duplex stainless steels, and Ni-base alloys, depending on corrosion environments:

(1) Decomposition of inhibitors by elevated temperatures at the well bottom
(2) Increase in operation cost due to inhibitor injection

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Table 1 Mechanical and impact properties required for 13 Cr stainless steel OCTG

User	Grade	*YS* (ksi)	*TS* (ksi)	*El* (%)	Hard-ness (HRC)	Toughness (ft·lb) avg.	Toughness (ft·lb) min.
A	80	80～ 95	≧ 95	API Formula	≦23		
	85	85～100	≧100	API Formula	≦23		
B	13Cr-75	75～ 90	≧ 95	API Formula	≦22	30	23
	13Cr-80	80～ 95	≧ 95	API Formula	≦23	28	22
	13Cr-95	95～110	≧105	API Formula	≦26	25	20
C	L80 13Cr	80～ 95	≧ 95	API Formula	≦23		
D	80	80～ 95	≧ 95	API Formula	≦23		

in submarine oil gas wells

Especially, for a so-called sweet environment which contains CO_2 and Cl^- but not H_2S, 13 Cr stainless steel shows substantial corrosion resistance. The increase in the consumption of these grades which are inexpensive compared with other alloys has been conspicuous in recent years.

Kawasaki Steel has also been manufacturing, using the Mannesmann-mandrel mill, 13 Cr stainless steel tubes which are excellent in corrosion resistance to CO_2. A typical example of the characteristics required for 13 Cr stainless steel for oil wells is shown in **Table 1**. As is seen from the table, the demand for 75, 80, and 95 ksi grades is large. Of the hardness of these grades, the upper limit is often required because of the necessity of resistance to sulfide stress cracking (SSC).

3 Hot Workability of 13 Cr Stainless Steel

Mannesmann-mandrel mill rolling process is composed of three processes such as piercing, mandrel-mill rolling, and hot stretch reducing. When rolling 13 Cr stainless steel, problems are developed due to inside cracks during piercing process and tube end cracks and also due to cracks along the flange part which does not contact the mandrel bar when rolled on the mandrel mill.

In piercing process, a billet is pierced as it is rotated between one set of horizontal cross rolls and a plug. In this process, the billet is brought to contact the rolls prior to contacting the plug and is fed in the longitudinal direction as it is undergoing repeated tension and compression workings until it finally contacts the plug. Therefore, in piercing a billet whose hot workability is low, inside cracks will occur before the billet contacts the plug (called as Mannesmann effect), resulting in inside defects sometimes. In order to prevent this defect, contrivance is needed so as to allow the plug to contact the billet and start piercing before cracks take place by the Mannesmann effect. Thus, it is necessary to increase the amount of plug advance. However, it is noted that the large amount of plug advance will create a sticking failure. This failure takes place due to the fact that not only the stress distribution but also the thermal history between the outer part and the center part of billet are different. Namely, the temperature of the billet outer part which has been heated up to 1 250°C falls down to about 1 170°C during piercing, whereas the temperature drop of the inner part is small.

Since the rolling temperature at the mandrel mill, on the other hand, ranges from 900 to 1 100°C, it is necessary to raise the hot workability of 13 Cr stainless steel in the range of 900 to 1 250°C to reduce the defects caused by cracking.

The hot workability of 13 Cr stainless steel was studied by conducting high temperature tensile tests and cross rolling of billets without plug, using the test piercer. The high temperature tensile tests are to evaluate the hot workability of 13 Cr stainless steel when being worked by piercing and rolling with the mandrell mill, while the cross rolling of billets without plug by the testing piercer is to assess the resistance to cracking due to the Mannesmann effect.

3.1 High Temperature Tensile Test

Specimens with a typical chemical composition shown in **Table 2** are tested to study their hot workability using a Greeble high temperature tensile tester. Taking into consideration the thermal history of a billet during piercing, the specimens are subjected to tensile tests at various temperatures relevant to the transient temperatures during the cooling cycle after heating the specimens up to a temperature equivalent to the normal heating temperature of billets.

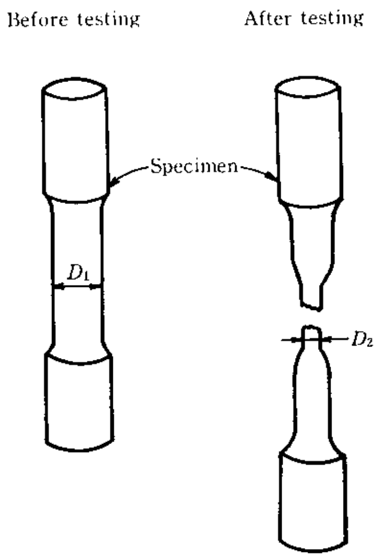
The specimens are solid piece as shown in **Fig. 1**, their workability evaluated by diameter reduction ratio before and after rupture.

Figure 2 shows the influence of sulfur content on hot workability. With a reduction of sulfur content, hot workability increased considerably, showing the values equivalent to those of carbon steel at temperatures of 1 000°C or higher when sulfur content reaches 0.001%. In the case of materials of high sulfur content, it is said that the hot workability is markedly deteriorated

Table 2 Standard chemical compositions of 13 Cr stainless steel

(wt %)

	C	Si	Mn	P	S	Ni	Cr	Mo	N
13% Cr	0.20	0.50	0.60	≦0.020	0.001	—	13.0	—	0.025



Reduction of diameter $= \frac{D_1 - D_2}{D_1}$

Fig. 1 Measurement of the O.D. reduction in the hot tensile test

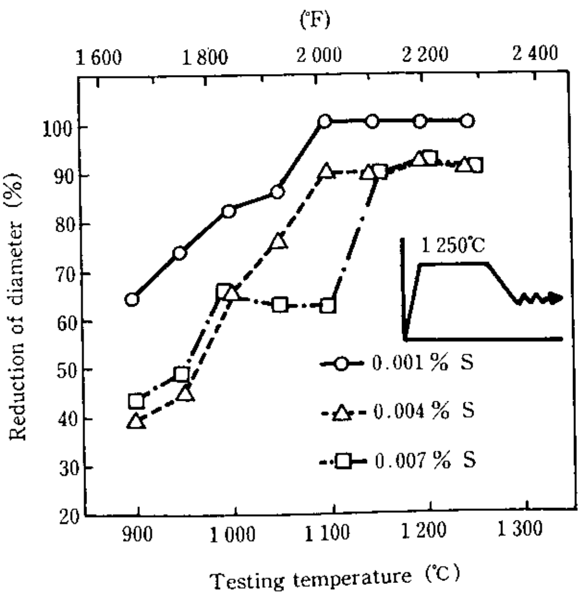


Fig. 2 Effect of S-content on hot workability of 13 Cr stainless steel

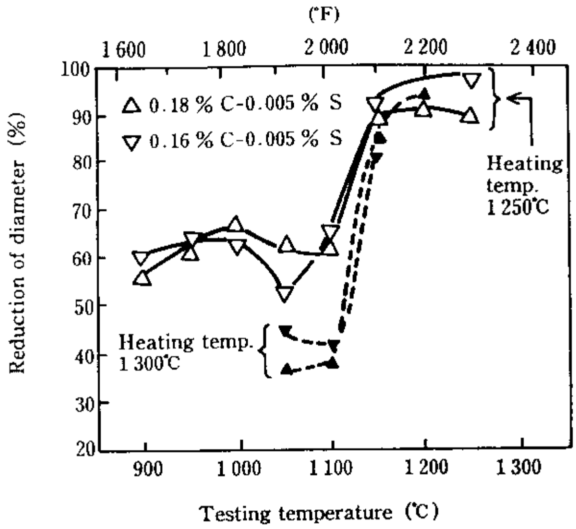


Fig. 3 Effect of heating temperature on hot workability of 13 Cr stainless steel

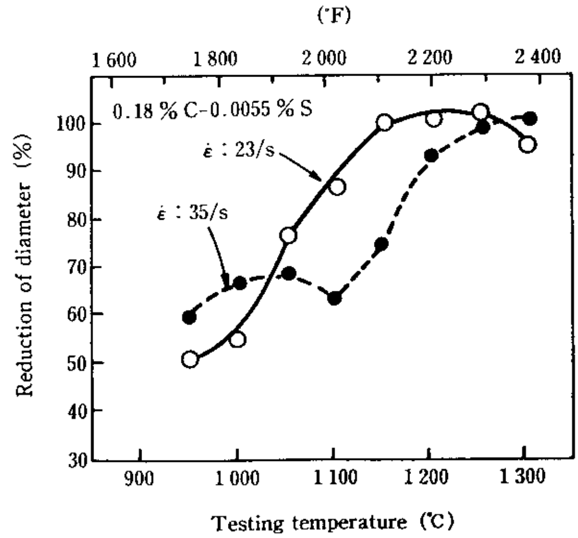


Fig. 4 Effect of strain rate on hot workability of 13 Cr stainless steel

because sulfides in solid solution, when heated up to a high temperature, precipitate at the grain boundaries during cooling.[1)]

The effect of heating temperature on hot workability is shown in **Fig. 3**. As is seen from this figure, the lower the heating temperature is, the better the hot workability is. This is because of the reason that the higher the heating temperature, the more increases the amount of sulfur to turn into solid solution for the second time from the existence as sulfides of Mn and Fe prior to heating.

The influence of strain rate is shown in **Fig. 4**.It suggests that the faster the strain rate, the worse the hot workability.

3.2 Investigation of Mannesmann Effect

Figure 5 shows the critical reduction within which the occurrence of cracks at the center part of the billet can be saved when it is cross rolled without plug by the testing piercer. With decreasing sulfur content, the critical

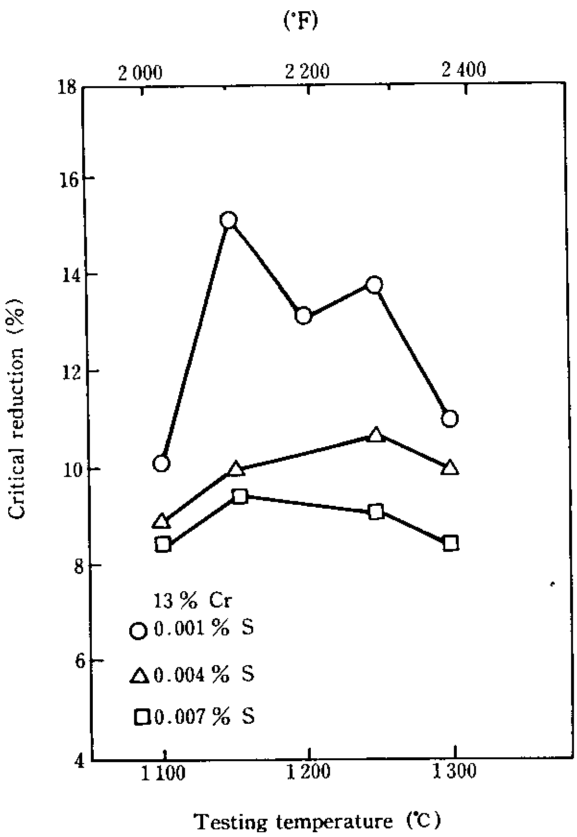


Fig. 5 Critical reduction to Mannesmann effect of 13 Cr stainless steel

reduction becomes larger, and the draft ratio (the reduction of billet) can be raised up to about 14% when sulfur content is 0.001%. This permits the flexibility of plug positioning and facilitates piercing operation.

4 Actual Tube Manufacturing Techniques

4.1 Manufacturing Process

4.1.1 Manufacturing of billets

The manufacturing process of 13 Cr stainless steel billets is charted in **Fig. 6**. The chemical composition of this material is the same as that shown in Table 2. The sulfur content of this material was lowered drastically on the basis of the results of the basic investigation on hot workability previously mentioned. For the round billets for seamless tubes, continuously cast material was used. Heating conditions and rolling schedules were determined by applying the basic findings relating to the hot workability previously discussed. To prevent cracks caused by the phase transformation from austenite to martensite, billets were slow-cooled after rolling.

4.1.2 Manufacturing of 13 Cr stainless tubes for oil wells

Figure 7 depicts a flow chart of the manufacturing process of 13 Cr upset tubings. The round billets, after piercing and rolling on the mandrel mill installed at the small-diameter seamless tube mill, were subjected to an annealing at a temperature below Ac_1 and ultrasonic flaw detecting tests. The tubes which passed inspection were put to upsetting and then quenched and tempered.

4.2 Rolling Techniques

When manufacturing 13 Cr stainless steel on the Mannesmann mandrel mill, the largest problem is cracks which occur on the outside and inside surfaces of tube. These cracks are ascribed to the low hot workability and the high hot deformation resistance of 13 Cr

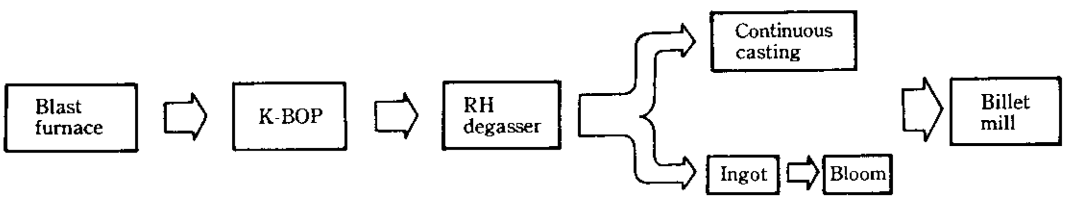


Fig. 6 Manufacturing process of 13 Cr stainless steel billet

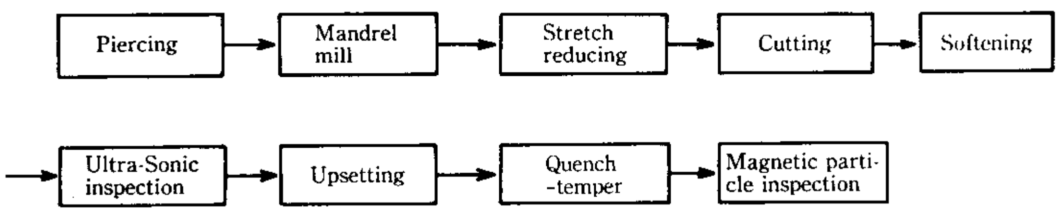


Fig. 7 Manufacturing process of 13 Cr stainless steel upset-tubing

stainless steel compared with those of carbon steels. As was discussed previously, sulfur content has been noticeably lowered to improve hot workability. The problem of high resistance to deformation, however, has still been left unsolved. The problems involved in rolling and their countermeasures will be discussed here based on the findings obtained from actual operation.

4.2.1 Rolling load

The hot deformation resistance of 13 Cr stainless steel, being 1.5 times that of carbon steel, bring about larger rolling reaction force at various mills. The rolling reaction of 13 Cr stainless steel at each mill is shown in **Fig. 8** by comparing with that of carbon steels containing 0.25% C. When rolling thin-walled tubes, not only the rolling load of the mandrel mill increases, but also the stripping capability of a mandrel bar becomes seriously worse. This is because of the tendency of the shell to become tightened compared with the case of carbon steels. This difficulty has been solved' by an improvement of the roll pass of the mandrel mill.

When rolling thick-walled tubes, on the other hand, the upper limit is determined by the power required for rolling by the stretch reducer. **Figure 9** shows the range of rolling capability of 13 Cr stainless steel of Kawasaki Steel obtained from the load on each mill and the capability of each mill.

4.2.2 Life of piercer tool

The piercing conditions of the piercer and the materials of the plug and the guide shoe exercise considerable effects on the surface quality of 13 Cr stainless steel

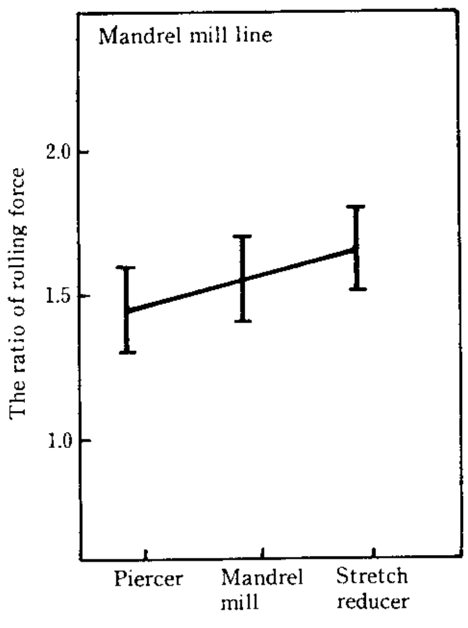


Fig. 8 The ratio of rolling force of 13% Cr stainless steel to that of 0.25% C steel

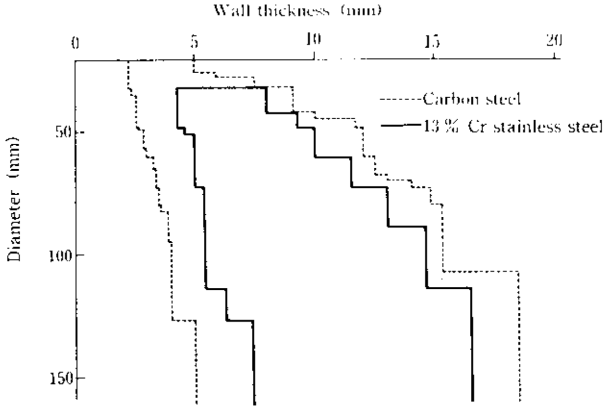


Fig. 9 Available size

tubes manufactured on the mandrel mill.

(1) Plug

In rolling 13 Cr stainless steel, its piercing efficiency is low due to its high resistance to deformation compared with that of carbon steels. The high deformation resistance and the prolongation of piercing time due to low piercing efficiency cause severe wear on the plug surface. To reduce this plug wear, the development of a tool material which is excellent in high temperature strength is necessary, not to speak of the importance of improving the tool configuration. Experimental plugs, to which such alloying components of solid solution strengthening type as W, Co, and Nb were added to raise high temperature strength, have been manufactured and subjected to wear tests by piercing. The results were shown in **Fig. 10**. Addition of alloying elements particularly increased the high temperature strength.

In actual operation, a plug with a chemical composition of specimen No. 2 in Fig. 10 was used. When piercing time was restricted to 10 s and under appreciable failures were observed on the plug surface even after the rolling of two billets.

(2) Guide Shoe

Piercing of 13 Cr stainless steel tends to cause wear and sticking to the guide shoes, because of the difference in the composition of scales on the outside surface of the piercing material in addition to the high deformation resistance and low piercing efficiency of the above material. This defect, being called as shoe mark, is not only responsible for the outside defects but also largely hampers productivity through an increased frequency of changing over of the guide shoes. To prevent this sticking phenomenon, ductile cast iron shoes are employed. The ductile cast iron shoes are excellent in resistance to sticking by the effect of spheroidal graphite included in the shoe material. These shoes, however, need earlier replacement because they have problems in

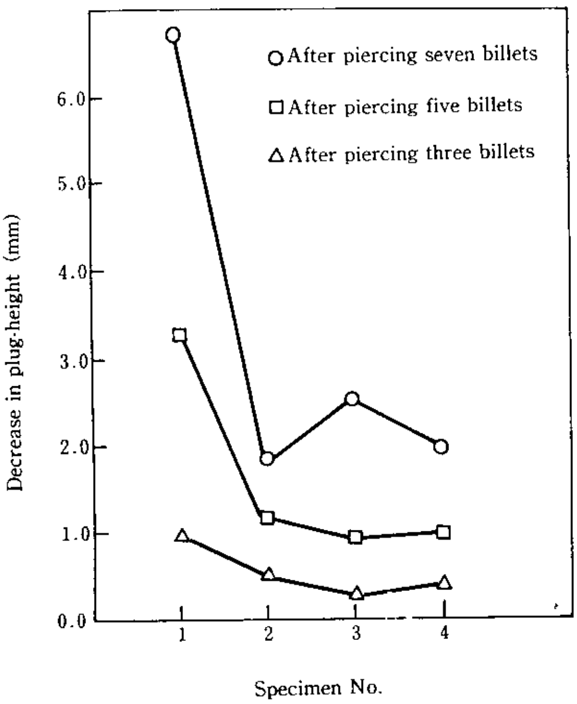


Fig. 10 Effect of chemical composition on wear-resistance in model piercing mill

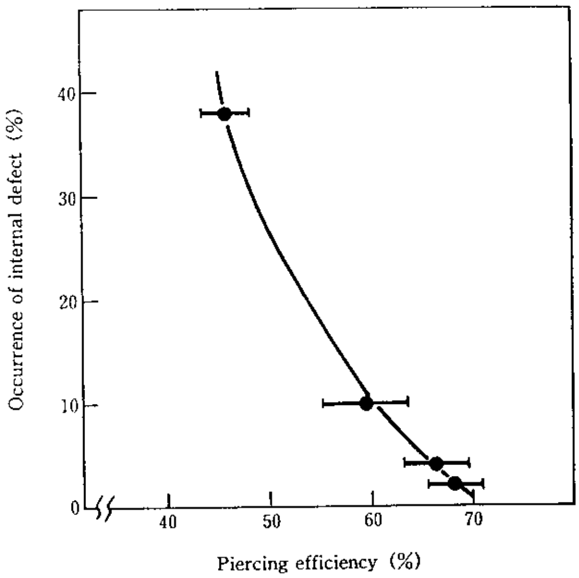
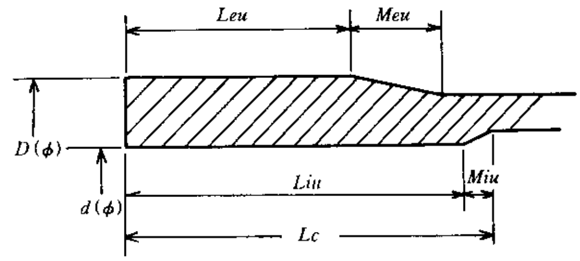


Fig. 11 Effect of piercing efficiency on internal defect



(mm)

	Long upset	Standard upset
Leu	≧149	≧101.6
Liu	≦274.8	≦224.0
Lc	≦304.8	≦254.0
Meu	≧28.58	
D	84.33~87.50	
d	55.44~58.67	

Fig. 12 Dimensions of upset-end

high temperature strength and noticeably worn out, their piercing efficiency lowering with an increased number of piercings.

4.2.3 Surface defects

The most frequent outside defects, when rolling 13 Cr stainless steel, are cracks which occur linearly in the direction of tube length, while, as to the inside surface, scab-like defects are most frequent. The occurrence of outside surface cracks is considered to be caused by the highly frequent repetition of shearing deformation when rolling billets with a low piercing efficiency.

This low piercing efficiency exercises adverse influences on the inside surface characteristics, as well. Because the number of cross rollings of billet without plug increases with a decrease in piercing efficiency, the frequency of occurrence of inside surface scabs increases as is shown in **Fig. 11**. Therefore, to raise the piercing efficiency with the piercer can be said to constitute an extremely important prerequisite in rolling high Cr steels.

4.3 Upsetting Techniques

4.3.1 Upset configuration

Figure 12 illustrates an upset configuration of the 13 Cr tubings which were manufactured this time. This is a heavy upset tubing with a total upset ratio of 177%, and the length of the upset part is 2″ longer than that of ordinary standard products. For this reason, the heating part of the tube end needs elongating, thus giving disadvantage to buckling. Such upsetting process therefore was feared to be considerably difficult.

4.3.2 Problems in upsetting

In working heavy upset products, the following three items may be given as factors that influences their quality:

(1) Upset Ratio per Shot
In view of a long heating length and hard workability, tubings were upset with an upset ratio reduced about 30% per shot. The upsetting schedule is on the basis of three heats and four shots.

(2) Pipe Heating Temperature
According to the results of the high temperature tensile tests shown in Fig. 3, workability decreases sharply at 1 150°C and under. Thus, the temperature at the tube end was aimed at 1 150°C and over when the tube was brought into the die set for upsetting.

(3) Die Lubrication
In heavy upsetting, especially in upsetting 13 Cr stainless steel, the quality of die lubricant is highly important. This is because the upset load applied at the tube end must entirely be directed to the deformation of the overall material without being absorbed in the friction between the tube and the dies.

The most useful lubricant currently used for high temperature working is graphite. However, enough lubricity will not be obtained by merely applying the graphite diluted with water. To allow the lubricant to exhibit its full lubricity, the diluent water of the lubricant must evaporate instantaneously when applied, leaving a tightly formed graphite film on the surface of the die. To insure this effect, die temperature adjustment is needed prior to the application of the lubricant. To find the optimum temperature which permits the formation of effective and tight enough lubricant film on the surface, the relation between the die temperature at the time of lubricant application and friction coefficient was studied using an abrasion tester. The lubricant used in this test was a water soluble graphite. As is shown in **Fig. 13**, there is a point in the vicinity of a lubricant-application temperature of 150°C where friction coefficient becomes lowest. With application of this finding to actual operation, dies were preheated and the optimum die temperature was insured.

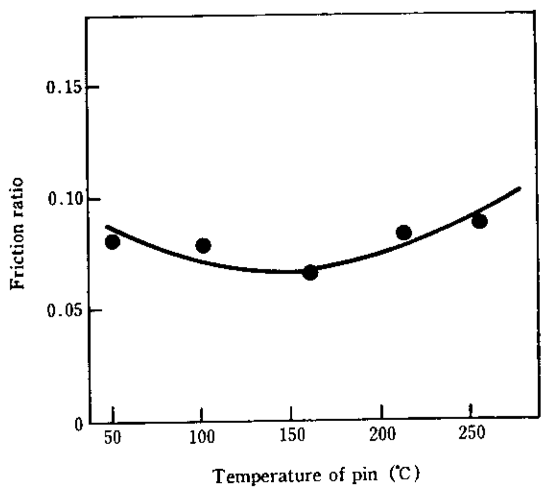


Fig. 13 Effect of temperature on friction ratio between pin and V block

5 Quality Characteristics

The quality characteristics of 13 Cr upset tubing 2-7/8" × 6.5 lb/ft (API 5ACL-80) manufactured this time are shown below.

5.1 Dimension and Shape

Figure 14 shows distributions of outside diameters, wall thicknesses, and eccentricities in a cross section of various tubes. They all fully satisfy API standards, being at the same level of ordinary carbon steel tubes for OCTG use.

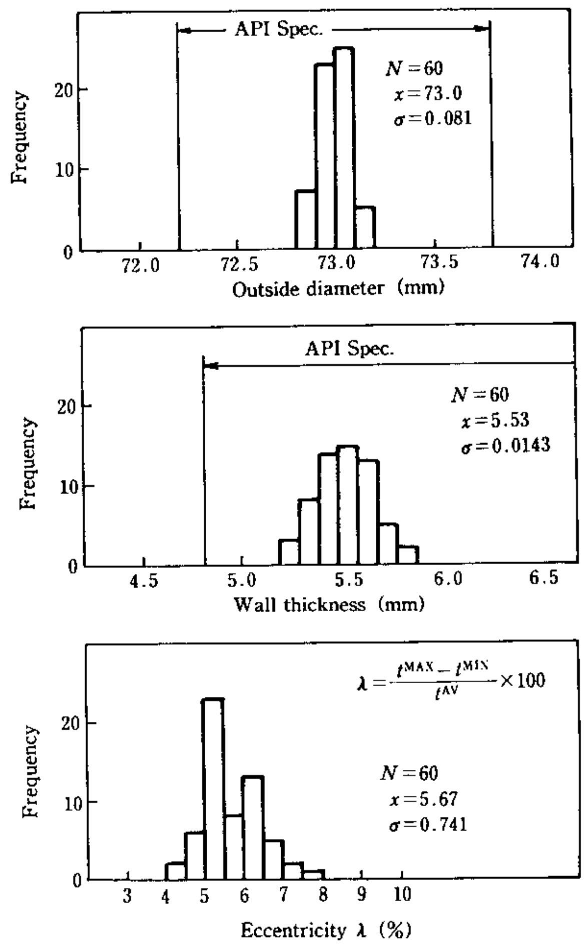


Fig. 14 Frequency distribution of pipe dimensions of 13 Cr stainless steel
(tube size: 73.0 mmϕ × 5.51 mm$t$)

5.2 Mechanical Properties

In order to determine quenching and tempering conditions for the operation of production equipment, tensile properties of 13 Cr stainless steel after tempering were investigated by conducting laboratory tests.[2] The quenching condition was to hold the specimen for 30 min at 970°C and then to air cool. Under this condition, the microstructures after quenching show mostly a martensitic structure. **Figure 15** shows the relationship between tempering parameters used for tempering of carbon steels and tensile properties. This figure indicates that controlling the tempering parameter in the range of 19 400 to 20 100 gives favorable results. Based on the laboratory test results, a system has been obtained whereby the strength of 13 Cr stainless steel can be estimated from its chemical composition and tempering parameter. The equation for tensile strength of the system mentioned above is expressed as Eq. (1):

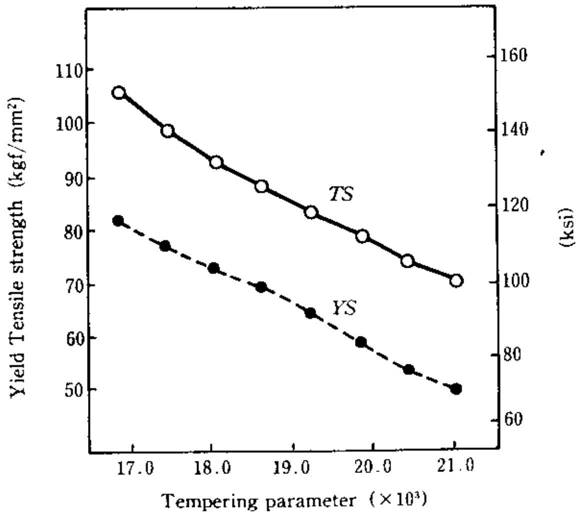


Fig. 15 Relation between tempering parameter and mechanical properties of heat treated 13 Cr stainless steel

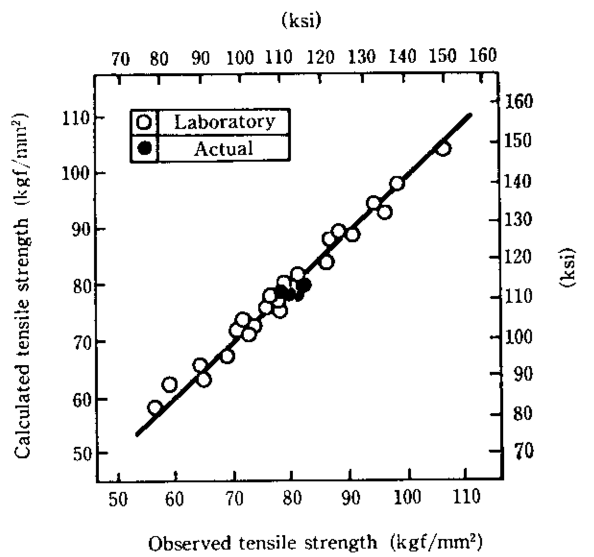


Fig. 16 Comparison between observed tensile strengths of heat treated 13 Cr stainless steel and calculated ones

Table 3 Production results of tensile properties of KO 13 CR-80 (2 7/8″ × 6.5 lb/ft)

	n	Yield strength (ksi)		Tensile strength (ksi)		Elongation (%)		Yield Ratio (%)	
		$\bar{x}$	σ	$\bar{x}$	σ	$\bar{x}$	σ	$\bar{x}$	σ
Pipe body	10	86.0	0.91	108.5	1.07	42.5	4.80	79.5	0.53
Upset part	18	86.9	2.93	110.6	1.99	33.7	1.86	78.6	1.99

$$\begin{aligned}\text{Calculated tensile strength} &= 3.416\sqrt{C+N} \times (T.P.)^2 \\ &\quad - 145.6\sqrt{C+N} \times (T.P.) \\ &\quad + 1\,642\sqrt{C+N} + 3.2Ni \\ &\quad + 5.7(Si - 0.5) + 33.1 \quad \cdots\cdots\cdots\cdots (1)\end{aligned}$$

$$T.P.\text{(tempering parameter)} = T(20 + \log t) \times 10^{-3}$$

T: Temperature (K)
t: Tempering time (h)

As is shown in **Fig. 16**, the estimated values obtained from Eq. (1) agree well with the measured values in the actual process.

The yield strength and tensile strength obtained by the quenching and tempering in the actual processing were shown in **Table 3**. There is no difference in strength between the upset part and the parent metal, and both of them are in full conformance with API 5ACL-80 standards. **Figure 17** shows the hardness distribution in the direction of tube length. Although the hardness is slightly high at the upset part, all of them satisfy HRC < 23, leaving no problem.

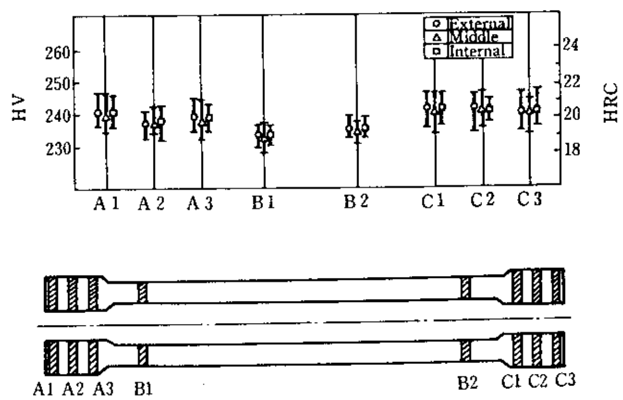


Fig. 17 Production results of hardness distribution of KO 13 CR-80 (2 7/8″ × 6.5 lb/ft)

5.3 Properties of Resistance to Corrosion

In environments where oil well tubings or line pipes are used, potential parameters which exercise significant influences on resistance to corrosion are; ① oil well temperature, ② CO_2 concentration, ③ Cl^- concentration, and ④ H_2S concentration. In this section, the corrosion resistance of 13 Cr stainless steel will be discussed in comparison with carbon steels in terms of the influences of temperature and CO_2 concentration. **Figure 18** shows the influence of solution temperature on corrosion rate in an atmosphere of 30 atm P_{CO_2} and a 3.5% NaCl solution. The corrosion rates of 13 Cr stainless steel in the temperature range from the normal room temperature to 100°C are extremely low compared with those of carbon steels and low alloy steels. At temperatures 150°C or higher, however, the difference of corrosion rate between 13 Cr stainless steel and carbon and low alloy steels becomes almost nil.

The reason that the corrosion rates of carbon or low alloy steels decrease at temperatures of 100°C and higher is because films such as stable magnetite or iron carbonate form on steel surface.

Next, the influence of CO_2 partial pressure on the corrosion rate in a 3.5% NaCl solution at 80°C is shown in **Fig. 19**. The corrosion rate of carbon and low alloy steels increases exponentially with increasing CO_2 partial pressure, while that of 13 Cr stainless steel is hardly influenced by CO_2 partial pressure. There are cases where materials come to rupture in an environment including H_2S at a stress which is extremely low compared with its yield strength. This failure is generally well known by the name of sulfide stress corrosion crack-

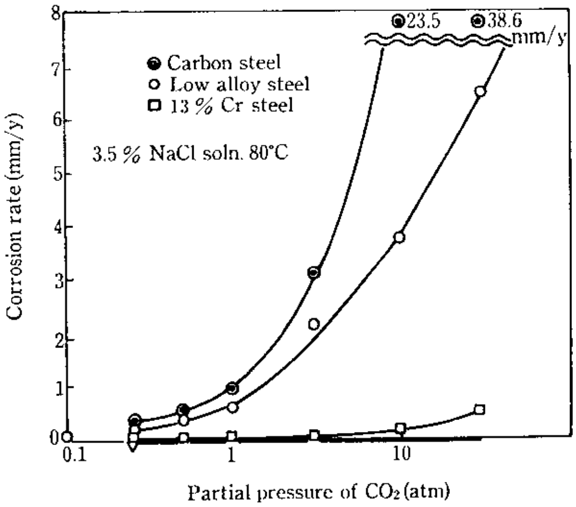


Fig. 19 Effect of CO_2 partial pressure on the corrosion rates at 80°C

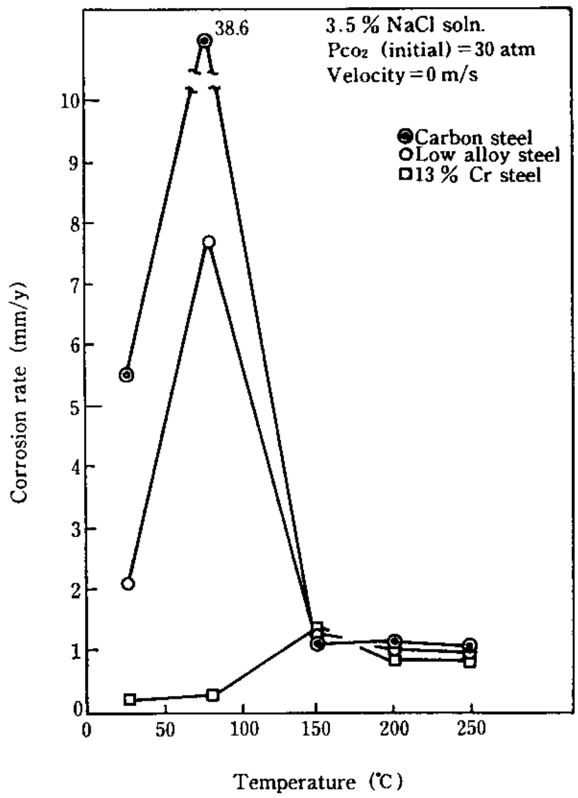


Fig. 18 Effect of temperature on the corrosion rate under 30 atm of CO_2

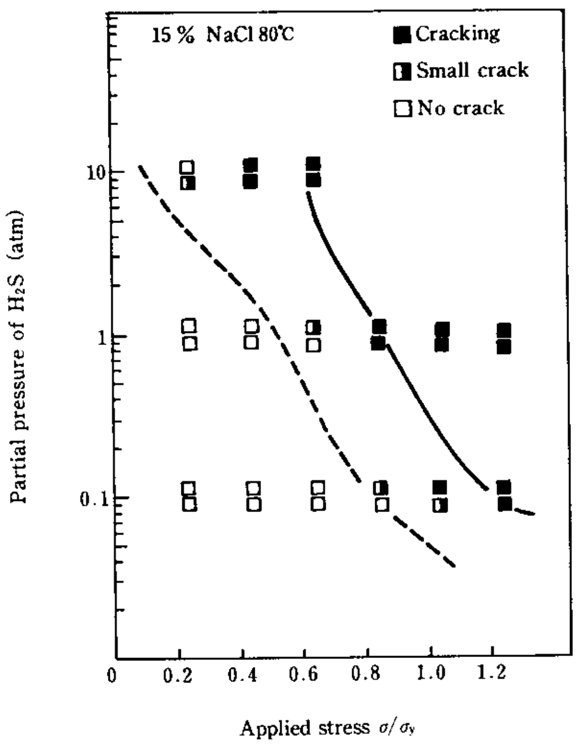


Fig. 20 Effect of applied stress and partial pressure on the SSC susceptibilities at 80°C

ing[3]. With increasing depth of oil well, the content of H_2S increases. This fact makes it important to select materials excellent in resistance to SSC. The influence of H_2S concentration on the resistance to SSC of 13 Cr stainless steel in a solution of 15% NaCl at 80°C is shown in **Fig. 20**. With increasing concentration of H_2S, 13 Cr stainless steel cracks at a low stress, which limits its usage in an environment including H_2S.

6 Conclusions

The following are a summary of the results of production techniques and quality characteristics of 13 Cr stainless steel tubings for OCTG based on the manufacturing results of 13 Cr upset tubings of 2-7/8" × 6.5 lb/ft (API 5ACL-80) which were manufactured this time.

(1) Manufacturing Techniques

(a) When rolling 13 Cr stainless steel by the Mannesmann process, it is important not to cause troubles in piercing process, and it is necessary to roll with high piercing efficiency to minimize defects on the inside and outside surfaces.

(b) The wear of plugs and guide shoes used for piercing of 13 Cr stainless steel is notable as compared with that for piercing of carbon steels. As to the plugs, the improvement in high temperature strength can be expected, while as to the guide shoes, the replacement with shoes made of ductile cast iron can be considered.

(c) As for upsetting, improvement of the lubricant for dies is needed. To attain this, a proper control of die temperature at the time of applying lubricant is needed.

(2) Quality Characteristics

(a) The dimensional accuracy acceptable to carbon steels can be fully satisfactory.

(b) The strength estimation equation obtained from the laboratory tests can predict the strength obtainable from quenching and tempering conducted in the relevant production process. The strength of 13 Cr stainless steel, which has undergone the quenching and tempering whose conditions have been determined based on the above mentioned equation, fully satisfies API L-80 standards.

(c) The corrosion rate of 13 Cr stainless steel in an environment at 100°C containing CO_2 and Cl^- is markedly low compared with that of carbon and low alloy steels, Under the same corrosion environment, the corrosion rate of carbon and low alloy steels exponentially increases with an increase of CO_2, whereas that of 13 Cr stainless steel is practically not affected by CO_2 partial pressure.

References

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