DEVELOPMENT OF A PRODUCTION TECHNIQUE FOR P/M ALLOY625 CLAD LOW ALLOY STEEL PIPE

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ABSTRACT

Powder metallurgy (P/M) coextrusion process for high alloy clad steel pipes was studied. Inert gas atomized Alloy 625 powder was extruded with extrusion ratios of between 1 and 12 to 1. Stable mechanical properties were obtained with an extrusion ratio of more than 3 to 1. Composite billets consisting of an Alloy625 powder layer and a wrought steel layer were semi-extruded to investigate the bonding behavior between these layers during extrusion. Penetration of the alloy powder particles into the wrought steel layer occurred at the initial stage of the extrusion. This phenomenon was found to be an advantage for the coextrusion of the clad materials.

Alloy625/ $2^1/_4$Cr-1Mo steel clad pipes were trially produced defect-free in practical production facilities through the P/M CIP-Extrusion process. These pipes exhibited appropriate mechanical properties, excellent corrosion resistance and especially excellent bond properties between the alloy liner and the steel substrate. Based on the obtained results, use of the P/M high alloy clad steel pipes as oil field line pipes and boiler tubes can be recommended.

INTRODUCTION

Highly corrosion resistant and high strength tubular materials are greatly demanded for the use of such as oil field line pipes and heat exchanger tubes in chemical plants. Cladding corrosion resistant alloys on high strength tubular materials is a rational way to achieve these multiple demanded features in one piece of pipe. The clad materials also provide an economical solution compared with high alloy monolithic materials.

The studied clad pipes consisted of Alloy625 and high strength low alloy steel. Although this kind of clad pipes

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have several benefits as composites, production of these pipes had been difficult. This was because of the poor workability of the high alloy materials and the great discrepancy of formability between the high alloy materials and the low alloy steels.

Recently studies on powder metallurgy have been accelerated owing to the availability of the inert gas atomized clean powders. These powder particles are rapidly solidified in the atomization process so that they have extremely low segregation and very fine microstructures. The fine grains in their microstructures improve workability of the materials and properties of the products. Usually Ni-base high alloys have poor workability due to high content of the alloying elements such as Cr, Mo and Nb. However they obtain extra workability in their rapid solidified powder forms due to the fine microstructures and low segregation. Considering these eminent advantages of the P/M process, application of the powder extrusion for production of the high alloy clad steel pipes, which has never succeeded through other methods, was tried.

Research works were initiated to establish a production technique for the high alloy clad steel pipes through coextrusion of the alloy powder and wrought steel. Basic study were carried out to investigate the consolidation of Alloy625 powder and the bonding process of the powder and the steel substrate during the coextrusion. Based on these studies, Alloy625/ $2^{1}/_{4}$Cr-1Mo steel clad pipes were trially produced. Evaluation on the properties of these clad pipes including mechanical properties, corrosion resistance and the bond properties were conducted.

MATERIALS and PROCEDURES

The Alloy625 powder was produced by nitrogen gas atomization and screened to -60 mesh. The chemical composition, the particle size distribution and the typical particle shape are shown in Table I, Table II and Fig.1 respectively.

Table I Chemical Composition of the Alloy625 Powder (wt%)

	C	Si	Mn	P	S	Cr	Mo	Nb	Fe	Al	Ti	Ni	O	N
Check	0.004	0.45	0.47	0.002	0.002	21.1	8.84	3.66	3.45	0.06	0.005	59.5	0.009	0.042
UNS N06625	<0.015	<0.5	<0.5	<0.015	<0.015	20.0 - 23.0	8.0 - 10.0	3.15 -4.15	<5.0	<0.4	<0.4	>58.0		

Table II Size Distribution of the Alloy625 Powder(um,wt%)

250/149	149/105	105/74	74/63	63/44	-44
14.9%	14.1%	16.7%	7.3%	13.9%	32.6%

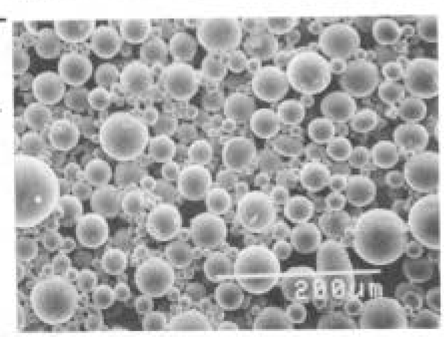


Fig.1 SEM micrograph of the powder

The Alloy625 powder was hot extruded to clarify the consolidation characteristics of the powder particles. The powder was poured into containers measuring 67 mm in diameter and 2 mm in thickness which were made of mild steel. After the containers were evacuated, sealed and heated up to 1423K for 1800 sec., the billets were extruded with extrusion ratios of between 1 and 12 to 1. After the extrusion, the materials were heated up to 1473K for 600 sec. and then water quenched. Tensile tests, impact tests and microscopic examinations were carried out to evaluate the influence of the extrusion ratio on the mechanical properties and the micro structures of the extruded alloy materials.

The composite billets which consisted of the alloy powder and a wrought carbon steel were prepared for the coextrusion. The structure of the composite billet is shown in Fig.2 which consisted of the wrought carbon steel layer measuring 67 mm in the outer diameter and 47 mm in the inner diameter and a thin mild steel capsule measuring 34 mm in the inner diameter and 2 mm in thickness. The annulus between the carbon steel layer and the mild steel capsule was filled with the alloy powder. The composite billets were extruded after heated up to 1423k for 1800 sec. with an extrusion ratio of 6.7 to 1 into clad steel pipes measuring 40 mm in the outer diameter and 32 mm in the inner diameter with the high alloy layer of 0.7mm in thickness. In order to investigate the bonding behavior between the high alloy powder layer and the wrought steel layer, the extrusion of the composite billets was halted in the middle of the process. This semi-extrusion experiment and the cross section of the test piece are shown schematically in Fig.3. Three different zones related their different deformation conditions are obviously identified in the semi-extruded test piece. One of them is the upset zone (A) where, mainly owing to deformation of the surface region of the powder particle, the powder layer was densified. The second one is the plastic deformation zone (B) where both the powder layer and the wrought carbon steel layer suffered shearing deformation by extrusion through the die. The third one is the clad material after the die (C) where the extrusion has been completed. By examining these three zones of the semi-extruded test pieces, the coextrusion process of the composite billet was investigated.

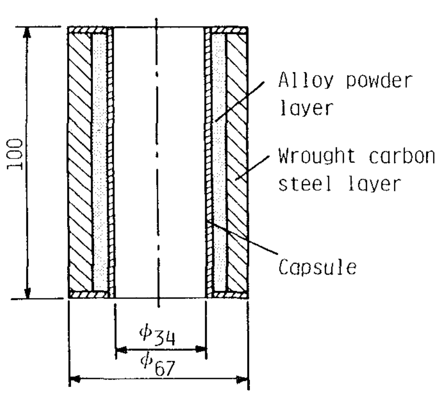


Fig.2 Composite billet

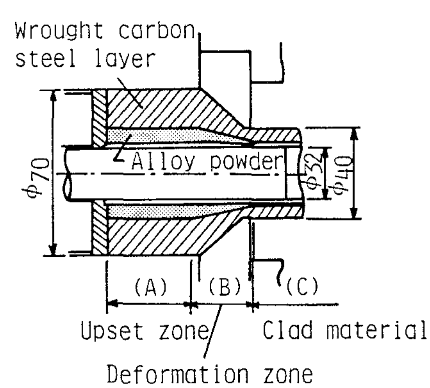


Fig.3 Outline of the semi-extrusion experiment

The Alloy625 clad $2^1/_4$Cr-1Mo steel pipes were trially produced in practical production facilities. Fig.4 shows a flow diagram for the P/M pipe manufacturing process with the CIP-Extrusion method (4). The qualified alloy powder was poured into the annulus of containers similar to the one shown in Fig.2. The low alloy steel pipe (i.e.,substrate) containing $2^1/_4$% Cr and 1% Mo formed the outer container component along with the concentric thin walled cylindrical piece of mild steel (i.e.,capsule). After the containers were sealed, the powder layer was densified to 75-85% of the full density by CIP. After heated up to 1423K, the composite billets were extruded with an extrusion ratio of 8.9 to 1.

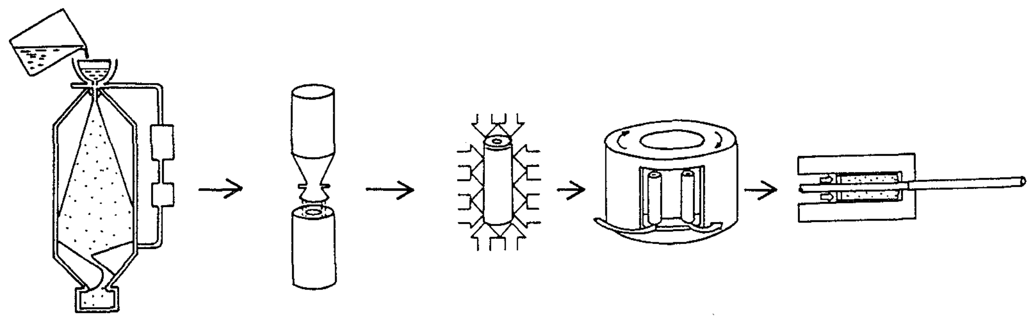


Fig.4 Flow diagram for the P/M pipe manufacturing process

While the low alloy steel pipe which was a part of the composite billet became one of the component of the finished product, the capsule was removed by pickling from the extruded pipes. The heat treatment conditions were selected so that the mechanical properties of the outer low alloy steel portion may achieve the specification of the API Grade X-75. The quenching and the tempering conditions were determined as follows.

Quenching:1253 K for 300 s followed by water quenching
Tempering: 973 K for 1800 s followed by air cooling

After the above shown heat treatment, strip specimens for the tensile tests and also notched bar impact test pieces of the Charpy type were sampled from the middle of the substrate wall. The long dimensions of the specimens was parallel to the longitudinal direction of the extruded pipes.

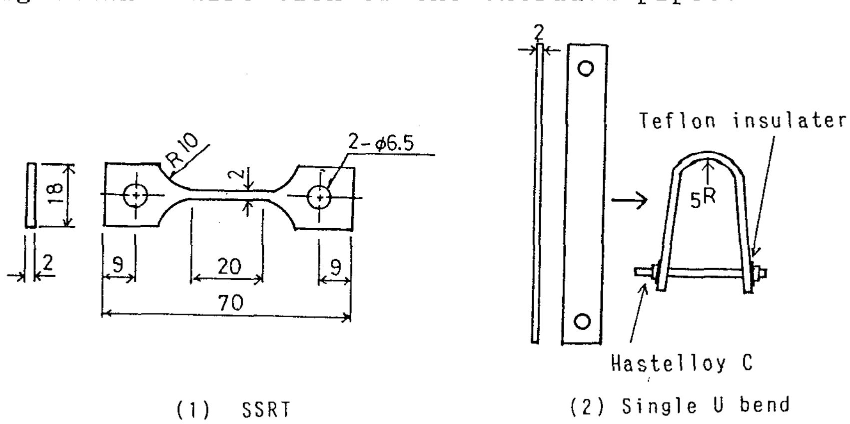


Fig.5 Specimens for the corrosion tests

The corrosion resistance of the liner material was evaluated in simulated sour gas environments. Slow strain rate test(SSRT) and immersion test were conducted on the specimens shown in Fig.5.

RESULTS

Extrusion process for powders is totally different from other P/M consolidation processes in a sense that it provides a large deformation on powder particles under a protected atmosphere. From this point of view, an extrusion ratio as a parameter for amount of the powder particle deformation might have a significant influence on the properties of the extruded products.

The mechanical properties of the extruded alloy powder with various extrusion ratios are shown in Fig. 6. Also the properties of a material which was simply consolidated by pressing the same powder until it reached its full density is shown as a material of an extrusion ratio of 1 to 1. As shown in Fig.6, while the strength are almost same irrespective of the extrusion ratio, the impact test values became slightly lower as the extrusion ratio decreases. According to these results, stable mechanical properties of the extruded P/M material were able to be acquired with an extrusion ratio of greater than 3 to 1.

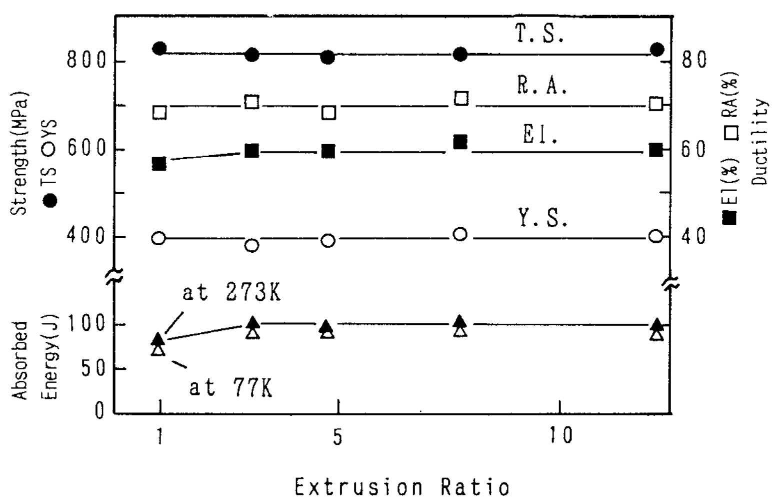


Fig.6 Results of the tensile tests and impact tests of the extruded P/M Alloy625 with various extrusion ratios

Ohashi et al.(2) had investigated the reason of the improvement of the ductility of extruded materials by the microscopic observations. They reported that more precipitates on the powder particle surface were separated as the extrusion ratio increases, which allows grain growth beyond the prior particle boundaries. This grain growth indicates that the large deformation caused by the extrusion has a significant effect on the powder particle bonding. The improvement of the impact properties with a higher extrusion ratio seems to be attributable to the same reason.

Since the bond properties between the liner material and the substrate are important, investigations on the bonding behavior between the powder layer and the steel substrate was carried out. Fig. 7 shows the micrographs at the interface between the alloy powder layer and the steel layer in the semi-extruded test piece. As shown in Fig. 7(A), the apparent density of the powder layer is about 80% at the initial stage, it has already reached 100% at the upset zone. What is more important in this zone is the phenomenon that a part of the alloy powder particles penetrate into the wrought steel surface. This phenomenon seems to be typical in this P/M process. Since the surface oxide films on materials possibly separated due to the powder penetration, it might provides a firmer interface bond for clad materials.

While the material was extruded through the die as seen in the deformation zone, Fig.7(B), the clad interface was extended and became rather flat as shown in Fig.7(C).

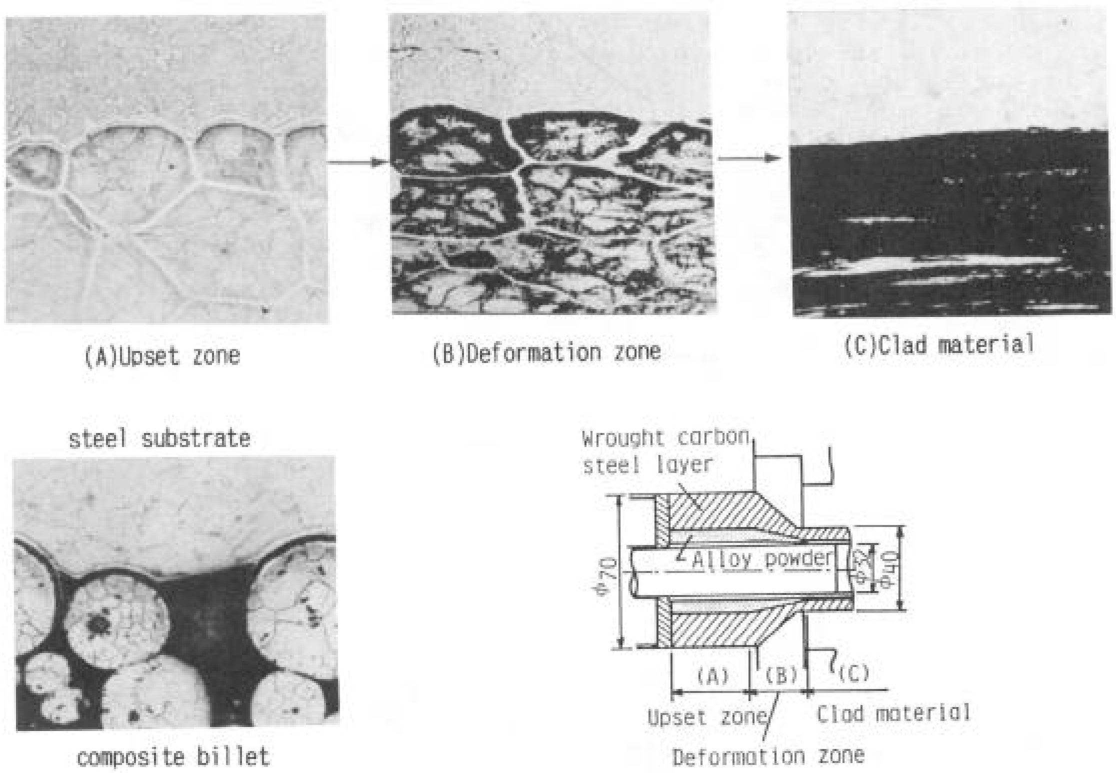


Fig.7 Interface bonding behavior in the coextrusion process

According to the test results mentioned above, the application of the P/M coextrusion process for the manufacture of the high alloy clad steel pipes seemed extremely successful. Therefore further investigation was conducted on the trially produced clad pipes to evaluate their properties. The pipes ,which consisted of the inner Alloy625 liner and the outer $2^{1}/_{4}$Cr-1Mo steel substrate, were manufactured in the practical production facilities after selecting the proper manufacturing conditions.

Fig.8 and Fig.9 show the macro structure and the interface microstructure of the trially produced clad pipe respectively. The chemical compositions of the liner and the substrate are listed in Table III. Table IV shows the typical tensile properties at room temperature and the impact properties on the $2^1/_4$Cr-1Mo steel portion(substrate). These results indicate that the P/M Alloy625 clad steel pipe has the appropriate mechanical properties.

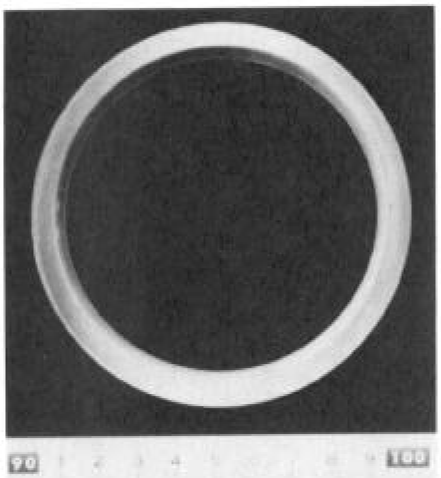


Fig.8 Macro structure of the trially produced P/M clad pipe

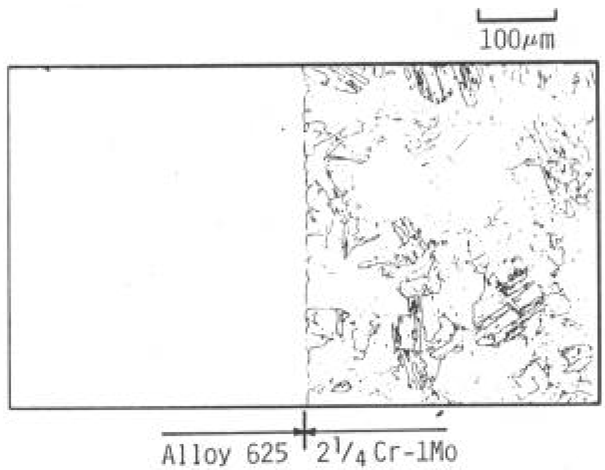


Fig.9 Interface microstructure of the trially produced P/M clad pipe

Table III Chemical Compositions (wt%)

	C	Si	Mn	Ni	Cr	Mo	Nb	Fe	O	N
Liner A625	0.005	0.45	0.47	59.7	21.0	8.88	3.63	3.46	.01	.045
Substrate $2^1/_4$Cr-1Mo	0.079	0.25	0.40	0.03	2.1	0.89		bal.		

The test results of the interface bond evaluation are shown in Table V. These results indicate excellent properties of the interface bond between the inner Alloy625 liner and the outer $2^1/_4$Cr-1Mo steel substrate.

Table IV Mechanical Properties

(1) Tensile test

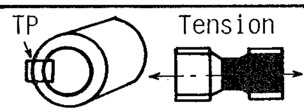
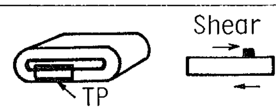
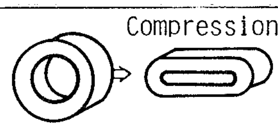
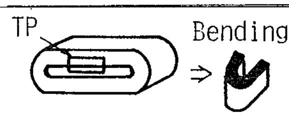
	0.2% Offset proof stress (MPa)	(ksi)	Tensile strength (MPa)	(ksi)	Elongation (%)
	624	90.7	719	104	23
Grade X-75		≥75		≥100	≥17

Table IV Mechanical Properties (continued)

(2) Impact properties (5mm x 10mm x 2mmV)

273 K		243 K	
(J)	(ft-lbs)	(J)	(ft-lbs)
114	83.9	105	77.4

Table V The Results of the Interface Bond Evaluation

Item	Test Pieces	Results
Interfacial Tensile Test		Tensile strength 725 MPa
Shear Test		Shear strength 425 MPa
Flattening Test		No interface debond No cracks
Bending Test		No interface debond No cracks

The SSRT results on the inner liner are shown in Table VI. The liner Alloy625 exhibited the similar tensile properties in the hostile environment as for in the pure water. Also, no secondary crack was observed after the test. The immersion test results are shown in Table VII. No pitting nor SCC (stress corrosion cracking) were observed on the liner Alloy625 after 4320 Hr immersion.

It has been clarified that there is little possibility of the hydrogen disbonding at the clad interface in case of subsea line pipes under the normal cathodic protection(3).

Table VI SSR Test Results at 400°F

Inert		Hostile *)			Ratio	
TTF(Hr)**)	RA(%)	TTF(Hr)	RA(%)	SCC	TTF(Hr)	RA(%)
35.1	52.0	35.6	53.7	No	101	103

*) 400°F 7atm H_2S, 25%NaCl+0.5%CH_3COOH, $\dot{\varepsilon}=4 \times 10^{-6}$

**) TTF: time to failure

Table VII Result of Single U-bend SCC Test

Temp (°F)	(°C)	H_2S (atm)	CO_2 (atm)	Solution	Time (Hr)	SCC
350	177	10	10	25%NaCl+	4320	No
450	232	10	10	0.5%CH_3COOH	4320	No

It is concluded that the trially produced P/M Alloy625 clad low alloy steel pipes had satisfactory corrosion resistance for line pipes in sour gas environments.

DISCUSSION

It is known that the coextrusion of these clad materials which have large discrepancy in flow stress requires severely restricted processing conditions. If they are not proper enough, the extruded materials may show periodical thickness deviations or cracks. The influence of several coextrusion factors on the workability of bimetallic materials have been calculated by Matsushita et al.,(1) through the energy method.

The coextrusion factors which affects workability of bimetallic materials are as follows; a flow stress ratio and a volume ratio of the materials, a die angle, an extrusion ratio, lubrication condition and a shear coefficient at the materials interface. The shear coefficient represents the difficulty of the slide which may occur between the adjacent metal layers. The coefficient m has a value between 0 to 1; if m=0, the layers slide with no friction and if m=1, layers have a perfect bond and no slide happens along the interface.

Fig.10 shows the effect of the shear coefficient m on the maximum possible extrusion ratio r when the coextrusion of a bimetallic pipe which has hard material inside are carried out. Where k denotes the flow stress ratio of the materials. The parameters used in the calculation are as follows; the volume ratio of the inside hard material : 0.29, the die half angle : 60 degrees, the friction coefficient between the die and the billet: 0.04. To simplify the calculation, the effect of the thin mild steel capsule was neglected.

According to Fig.10, the maximum limit of the extrusion ratio increases as the shear coefficient increases. This means if the two materials are difficult to slide against the other along the interface, the coextrusion becomes easier.

As mentioned before, the penetration of the powder particles into the wrought steel surface occurs on the initial stage of extrusion. This phenomenon prevents the sliding of material against the other. Consequently the phenomenon increases the maximum limit of extrusion ratio, which means the P/M process improves the formability of the clad materials.

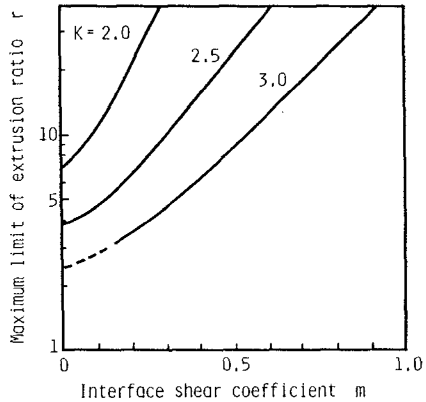


Fig.10 Effect of the interface shear coefficient on the coextrusion of clad materials

CONCLUSION

1. The production techniques for the high alloy / low alloy steel clad pipes were developed applying the P/M coextrusion process.

2. A trial production was conducted for the Alloy625/ $2^1/_4$Cr -1Mo steel clad pipes. Those clad pipes were proved to have appropriate mechanical properties, excellent interface bond properties and excellent corrosion resistance.

3. The penetration of the powder particles into the steel surface which occurred on the initial upset stage of the coextrusion seems to improve the formability of the coextrusion of the clad materials due to the increase of the shear coefficient between the material along the interface.

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(3) S.Azuma et al.,"Corrosion and its countermeasure of subsea CRA clad line pipe", CORROSION/90,Paper No.69, NACE (1990).

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