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Geometric Effect of Ion Nitriding on the Nitride Growth Behavior in Hollow Tube

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The growth behavior of the nitride layer inside a long hollow tube with an intricate geometry was studied to find a way to enhance the uniformity of the nitride layer. The inner surface of steel tube of 30-mm inner diameter was machined to have corrugation depths ranging from 0.65 to 3.90 mm and corrugation widths from 1.10 to 13.2 mm. After the specimens were ion nitrided at 525 °C for 10 hr in 2.5 torr operating pressure, the thickness of the compound layer and the diffusion layer on the land and the groove was measured and analyzed according to corrugation depth and width. As the corrugation becomes deeper, the thickness of the compound layer on the land increases and that of the layer on the groove decreases. The thickness of the diffusion layer on the land and the groove depicts a similar tendency to that of the compound layer. As the corrugation becomes wider, the compound layer thickness on the land decreases and that of the layer on the groove increases to the contrary. Thickness variations in the diffusion layer on the land and the groove resemble those of the compound layer. The nitride growth characteristics on the corrugated geometry in ion nitriding was discussed in view of Hollow Cathode Discharge (HCD) effect, nitrogen concentration, and the probability of compound adsorption.

1. Introduction

SURFACE hardening of materials has been a persistent problem to materials scientists and engineers. One the popular processes to achieve hardening of a steel surface is nitriding, which has been in commercial use since the early 20th century.[1] The nitriding process eliminates subsequent quenching because of its comparatively low processing temperature of 450 to 570 °C, and the distortion and dimensional changes in the workpiece are lessened with nitriding compared to carburizing.[2] Conventional nitriding is customarily carried out in an atmosphere of partially dissociated ammonia, or in a cyanide-cyanate salt bath, which includes potential environmental hazards and difficulties in controlling the growth of the white layer.

A newer nitriding process was patented in 1931[3] and has been getting wide attention as an industrial alternative to the conventional gas or salt bath nitriding of steel.[4] The new process, called ion nitriding, is accomplished by using the energy of the glow discharge. This ion nitriding process is characterized by its shorter process duration, cleanliness, and in particular its complete nontoxicity, as well as the ease with which it controls the growth of the undesirable white layer.[5,6]

Most studies to date have been concerned with the evaluation of engineering variables such as the alloying elements, temperature, and duration of ion nitriding, along with gas mixtures and operating pressure.[7,8] Ion nitriding has been compared with the various nitriding processes from the standpoint of its economic viability.[9] Further development of ion nitriding was undertaken to suppress arcing by supplying pulse direct current instead of direct current.[10,11] Ion nitriding characteristics achieved with the pulsed glow discharge were studied from an academic view by Kwon *et al.*[12]

Production engineering demands for low production costs in addition to increased wear resistance and fatigue strength of workpieces were met by surface hardening. No systematic work was undertaken to investigate the nitride layer growth inside long cylinders with intricate geometric shapes. The present

Table 1 Chemical Composition of Specimens

Composition, wt%							
C	Si	Mn	P	S	Cr	Mo	Fe
0.43	0.24	0.67	0.026	0.024	0.98	0.24	Bal

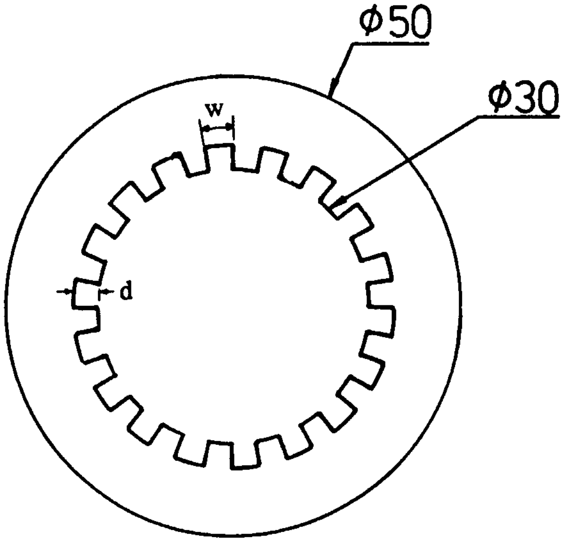

Fig. 1 Schematic of corrugations inside hollow cylinder.

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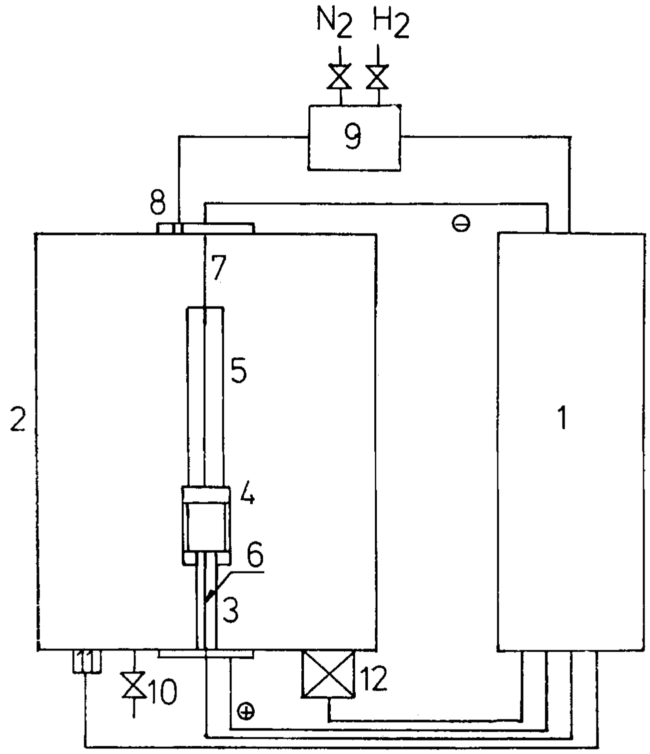


Fig. 2 Schematic of pulse ion nitriding system. (1) Control part and power supply. (2) Chamber (anode): ground. (3) Cathode. (4) Specimen holder. (5) Specimen. (6) Thermocouple. (7) Auxiliary anode. (8) Gas inlet. (9) Gas control part. (10) Leak valve. (11) Pressure sensor. (12) Rotary pump.

research objective was to examine the growth behavior of the nitride layer over the corrugated internal surfaces in a long, hollow tube and to discover engineering design factors to enhance the uniformity of the nitrided layer.

2. Experimental Procedure

A commercial grade of Cr-Mo steel, whose chemical composition is given in Table 1, was used in this investigation. Specimens were bored and machined to hollow cylinders of 200 (L) by 30 (ID) by 50 (OD) mm size. The internal surfaces of the hollow cylinder were machined to produce corrugations, as shown in Fig. 1. The corrugation depths were varied from 0.65 to 3.90 mm, and the corrugation widths were changed from 1.10 to 13.2 mm. Table 2 summarizes the corrugation depths and widths of the workpieces.

The specimens were thoroughly degreased, descaled, washed, dried, and positioned on the cathode stage in the ion nitriding chamber, as shown in Fig. 2. A stainless steel rod of 6 mm in diameter was used as an anode and positioned along the hollow cylinder inside the specimen. Before ion nitriding the specimen, a mixture of nitrogen and hydrogen gas (1:1) was used after evacuation to clean the surface by sputter cleaning. The gas mixing ratio of nitrogen to hydrogen was converted from 1 to 4, and the operating pressure was maintained at 0.4 torr throughout. The pulsed power of 20 kW was used in this study, and the voltage of the discharge was 500 V. All the samples were nitrided at 525 °C for 10 hr. The temperature of the workpiece was measured with a Chromel-Alumel thermocouple embedded in the cathode stage. The glow discharge power supply was cut off at the end of the treatment time, and the specimen was left to cool in vacuum.

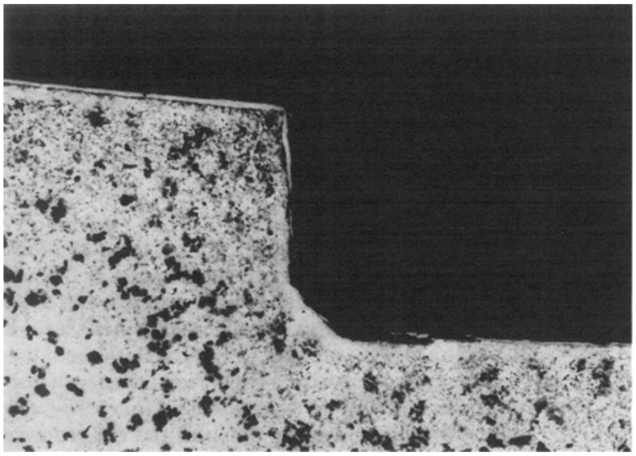


Fig. 3 Microstructure of the nitride layer on the land and on the groove of corrugations. (50×).

Table 2 Depth and Width of Specimen Corrugations

Specimen No.	Depth (*d*), mm	Width (*W*), mm
1	0.65	2.35
2	1.30	2.35
3	2.60	2.35
4	3.90	2.35
5	0.65	1.10
6	0.65	4.60
7	0.65	6.90
8	0.65	13.2

After ion nitriding, the specimens were sectioned circumferentially with a cutting wheel at a distance of 10, 50, 100, 150, and 190 mm from the bottom. Each sectioned piece was mounted, metallographically prepared, and etched with 3% nital. The compound layer thickness was determined with an optical microscope equipped with a micrometer. The thickness of the diffusion layer was determined by measuring from the surface to the point where the hardness value was 10% higher than the substrate, as determined from the hardness profile curve.

3. Results and Discussion

3.1 Thickness Variations in the Nitride Compound Layer Covering the Corrugated Surface

Figure 3 shows the typical microstructure of the nitrided layer on the land and on the groove of the corrugation. The nitrided layer is thicker on the land than on the groove. When the geometry was considered, the thickness decreased, in order,

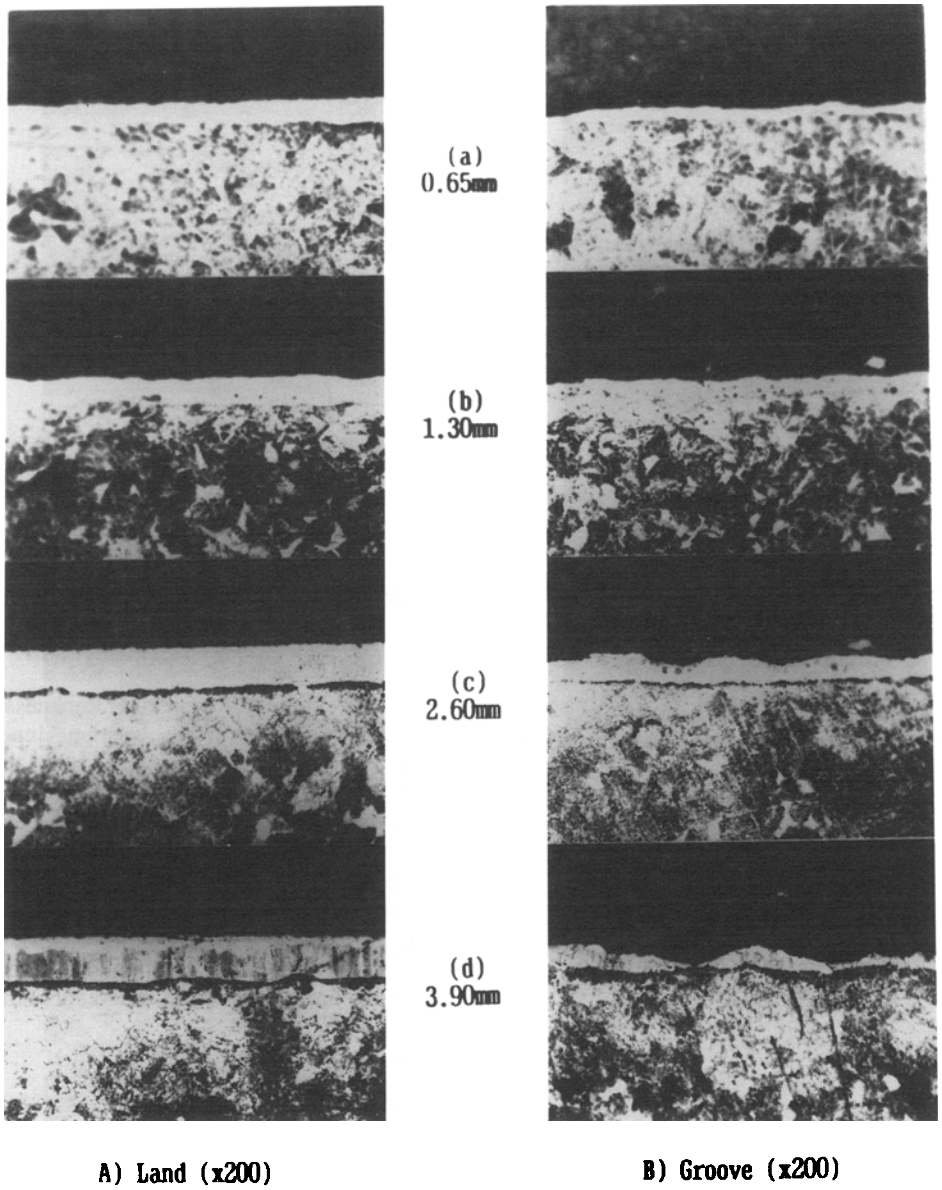


Fig. 4 Microstructure of the nitride layer on the land (A) and on the groove (B) of various corrugation depths.

Table 3 Variation in the Thickness of the Nitride Compound Layer on the Land and Groove with Corrugation Depth

Corrugation depth, mm	Thickness of nitride compound, μm Land	Groove
0.65	21	14
1.3	23	13
2.6	30	12
3.9	32	10

from the edge of the land, to the land, to the groove, to the edge of the groove. Variations in the compound thickness were found to depend on the Hollow Cathode Discharge (HCD) effect,[6] nitrogen concentration,[9] distribution of the electric field, and the probability of compound adsorption.[9] The above factors, except the distribution of the electric field, explained the variation in nitride thickness distribution over the corrugation inside the hollow cylinder. The effect of the electric field on the growth of the nitride layer is less significant than the resultant effect of HCD, nitrogen concentration, and the probability of compound adsorption.

3.2 Effect of Corrugated Depth

Figure 4 shows the microstructure of the land and the groove at the middle point of the specimen at various corrugation depths. The thickness of the nitride compound layer on the land and on the groove was measured, and the results are summarized in Table 3. As the depth of corrugation increased, the thickness of the compound on the land increased from 21 to 32 μm, and the thickness of the layer on the groove decreased from

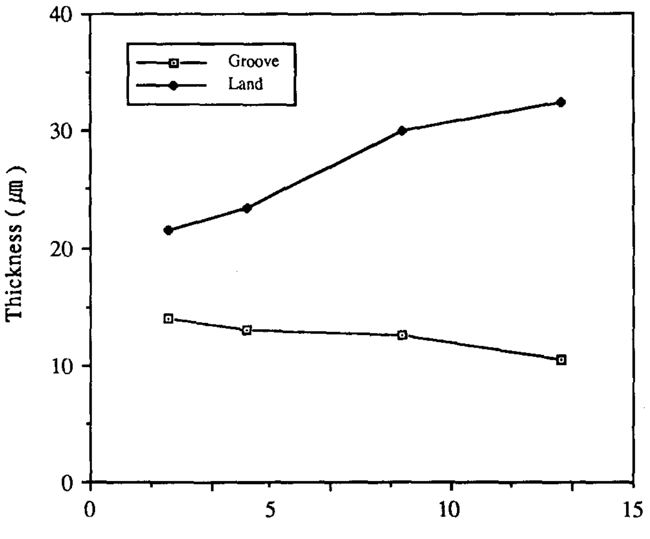


Fig. 5 Variation in the thickness of the nitride compound layer with corrugation depth.

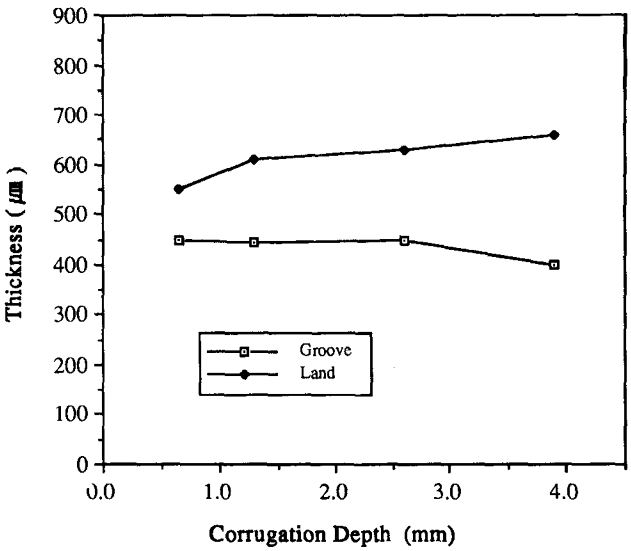


Fig. 6 Variation in the thickness of the nitride diffusion layer with corrugation depth.

Table 4 Variation in the Thickness of the Nitride Compound Layer on the Land and Groove with Corrugation Width

Corrugation depth, mm	Thickness of nitride compound, μm Land	Groove
1.1	24	13
2.35	21	14
4.6	19	15
6.9	19	15
13.2	17	16

14 to 10 μm. Variations in the thickness of the compound layer on the land and on the groove is depicted in Fig. 5. The thicker nitride layer growth on the land compared to the groove resulted from the fact that the groove experienced a greater HCD effect, lower nitrogen concentration, and a lower probability of compound absorption than the land. Figure 6 illustrates the diffusion layer thickness curve on the land, as well as on the groove, according to the depth of the corrugation. The resultant similar growth pattern of the diffusion layer compared to that of the compound layer generally agreed with that observed in a previous investigation.[13]

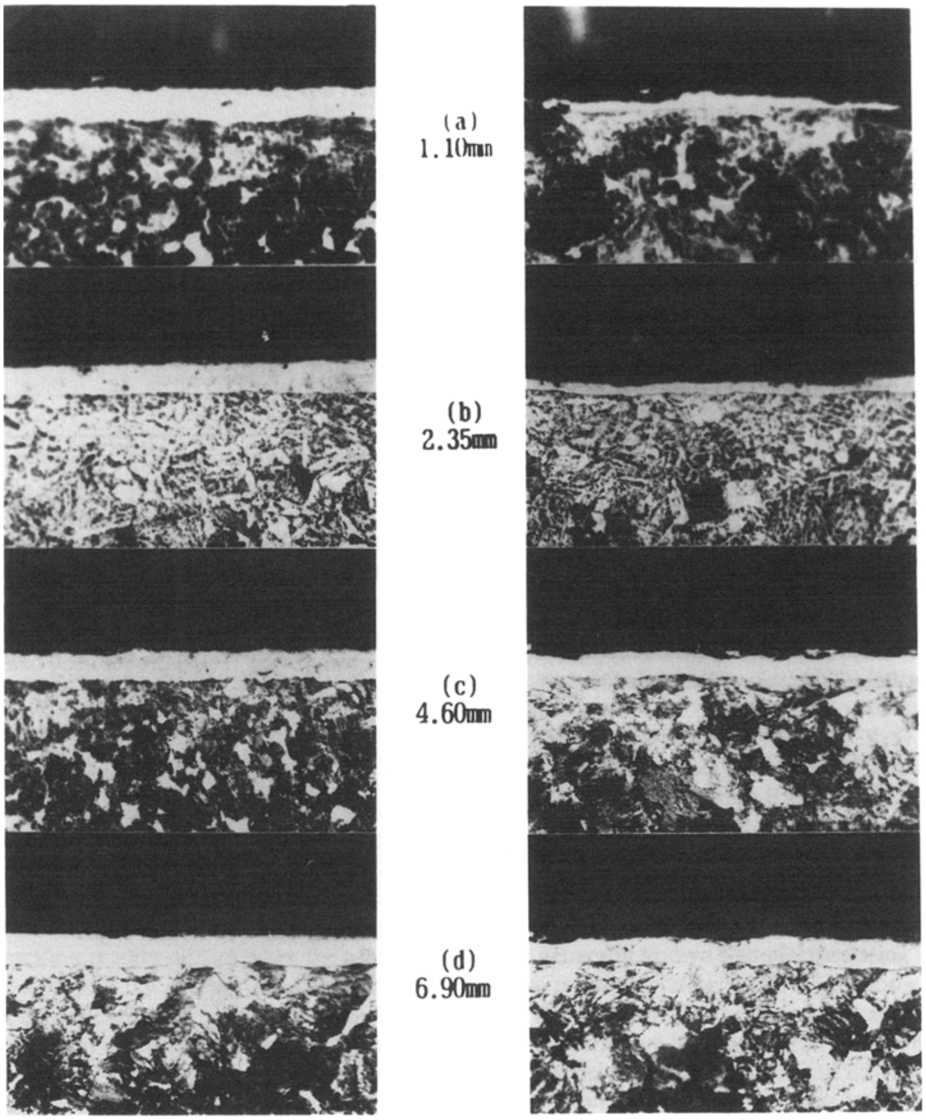
3.3 Effect of Corrugation Width

Figure 7 illustrates the microstructure of the land and groove at the middle point of the specimen at various corrugation widths. The thickness of the nitride compound layer on the land and on the groove were measured, and results are summarized in Table 4. As the width of the corrugation increased, the thickness of compound on the land decreased from 24 to 17 μm, and the thickness of the layer on the groove increased from 13 to 16 μm. Variations in the thickness of the compound layer on the land and on the groove are illustrated in Fig. 8, whereas the variations in the diffusion layer thickness are shown in Fig. 9. The nitride compound layer became thinner on the land, and the layer appeared thicker on the groove as the width of the corrugation increased. These two curves converged at the compound layer thickness of 14 μm as the width of groove increased to more than 13.2 μm. The probability of absorption of the compound appeared to be significant in the explanation of this phenomenon. It was conclusive that both the depth and the width of the corrugation contributed to geometrical complexity, which caused the growth of a compound and diffusion layer on the land and on the groove to be markedly different.

4. Conclusions

A Cr-Mo steel hollow tube with corrugations over the internal surface was ion nitrided for 10 hr at 525 °C in 2.5 torr operating pressure. The depth of the corrugations was varied from 0.65 to 3.90 mm, and the width of corrugation was varied from 1.10 to 13.2 mm. The following conclusions were made on the growth characteristics of the nitride compound layer and the diffusion layer based on the geometry of the specimen. As the depth of the corrugation increased, the growth of the nitride compound layer on the land increased, and the growth of the layer on the groove decreased. This phenomenon resulted from the HCD effect, the nitrogen concentration, and the probability of a compound adsorption. The growth of the diffusion layer on the land and on the groove exhibited a similar tendency to the growth of the compound layer on the corrugation.

As the width of the corrugation increased, the growth of the compound on the land decreased and on the groove increased, respectively. This was due to the significant effect of nitrogen concentration. Growth of the diffusion layer on the land and on the groove was similar to the nitride compound layer growth of the respective geometry.



A) Land (x200) B) Groove (x200)

Fig. 7 Microstructure of the nitride layer on the land (A) and on the groove (B) of various corrugation widths.

Both the depth and the width of the corrugation represented the complexity of geometry, which enhanced the differences in the growth of the nitride layer on the land and on the groove.

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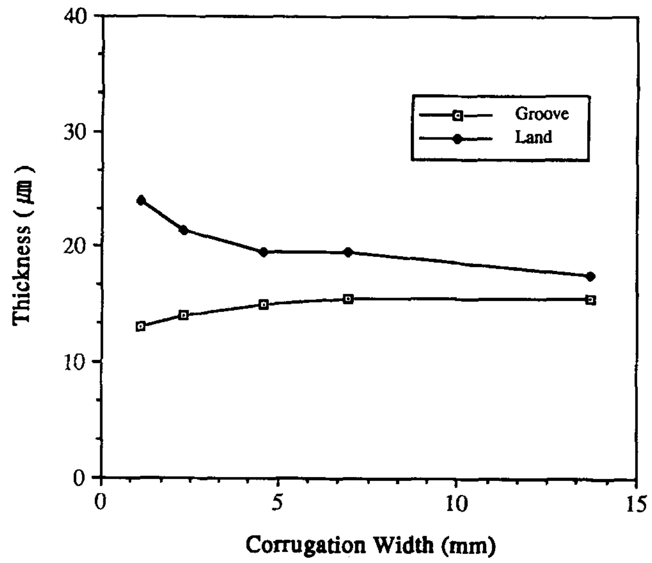

Fig. 8 Variation in the thickness of the nitride compound layer with corrugation width.

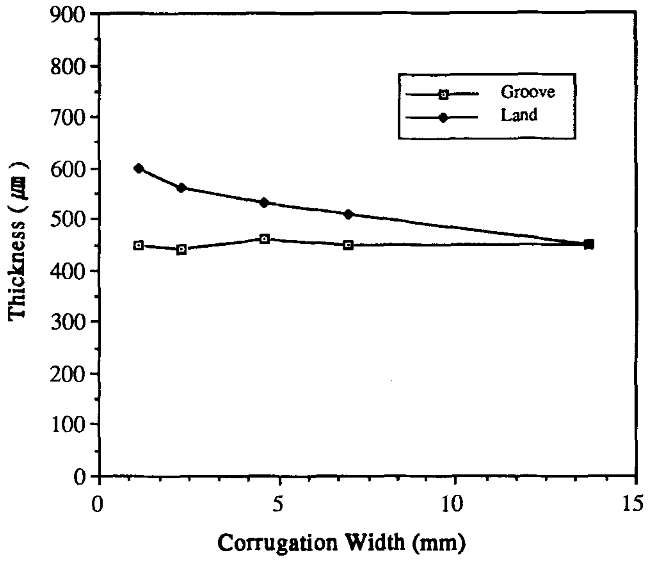

Fig. 9 Variation in the thickness of the nitride diffusion layer with corrugation width.

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