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Nitrocarburising in a Fluidiesed Bed Furnace With Co_2 Gas Additions (Studies on the properties of resulting compound layers)

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Abstrak

Karakterisasi terhadap lapisan senyawa hasil perlakuan panas feritik-nitrokarburisasi yang menggunakan dapur fluidised bed dan mengandung campuran gas CO_2 telah dilakukan di dalam penelitian ini. Penelitian menggunakan baja karbon AISI 1040 (0,4 %C) yang dinitrokarburisasi pada temperatur 570^0C, dimana komposisi atmosfir divariasikan sesuai penambahan 1-3% gas CO_2 dengan waktu proses 1-4 jam. Hasil pengujian XRD menunjukkan lampisan senyawa yang terbentuk prinsipnya terdiri dari γ' Fe_4(N,C) dan ε Fe_{2-3}(N,C). Meningkatkan kandungan gas CO_2 didalam atmosfir menyebabkan pembentukan fasa ε Fe_{2-3}(N,C) lebih stabil. Pengamatan metalografi dan XRD mengkonfirmasikan terbentuknya lapisan senyawa dengan fasa ε Fe_{2-3}(N,C) dominan untuk waktu proses 4 jam. Penambahan waktu proses dan kandungan gas CO_2 berkaitan dengan terbentuknya porositas pada lapisan senyawa. Hasil analisa SEM mengindikasikan porositas yang terbentuk tidak berlebihan. Sehingga dapat disimpulkan, nitrokarburisasi menggunakan dapur fluidised bed mengandung atmosfir 1-3% CO_2 dan waktu proses 4 jam dapat menghasilkan lapisan senyawa yang unggul terhadap keausan akibat gesekan.

Abstract

The characteristics of compound layers resulting from ferritic nitrocarburising with atmosphere containing CO_2 gas additions have been investigated using a fluidised bed furnace. The experiments made use of AISI 1040 steel. Treatment temperature was set at 570°C; atmosphere composition and treatment time were altered accordingly. Compound layers produced were essentially comprised of γ' Fe_4(N,C) and ε Fe_{2-3}(N,C). Increasing CO_2 contents and treatment time leads to stabilisation of ε phase and compound layer thickness. A predominantly ε phased layer was produced by 4 hours treatment duration. Porosity in the compound layer was found related with an increase in treatment time and CO_2 composition. At a present work. 4 hours treatment duration did not exhibit severity level of porosity. Therefore, it is concluded by the present experiment that nitrocarburising in a fluidised bed furnace with 4 hours duration and 1-3% CO_2 gas additions is capable to produce a superior anti scuffing compound layer.

Introduction

Ferritic nitrocarburising is a thermo - chemical surface treatment which is applied to a ferrous object in order to produce surface enrichment in nitrogen and carbon which forms a compound layer consisting of ε $Fe_{2-3}(N,C)$ phase. Beneath the compound layer there is a diffusion zone enriched in nitrogen. The treatment considerably enhances anti-scuffing characteristics, and in some instances the corrosion resistance of engineering components by means of the presence of a compound layer containing ε iron carbonitride. The fatigue characte - ristics of mild or low alloy steels are also improved when nitrogen is retained in solid solution in the diffusion zone due subsequent quenching from treatment temperature.

Ferritic nitrocarburising treatments were first introduced over forty years ago; since then an extremely wide range of engineering components, such as rocker-arm spacers, textile machinery gears, pump cylinder blocks and jet nozzles, have been treated for wear resistance, while components such as crankshaft and drive shafts have been treated for improved fatigue properties[1]. An example of automotive parts treated by nitrocarburising is shown in Figure 1.

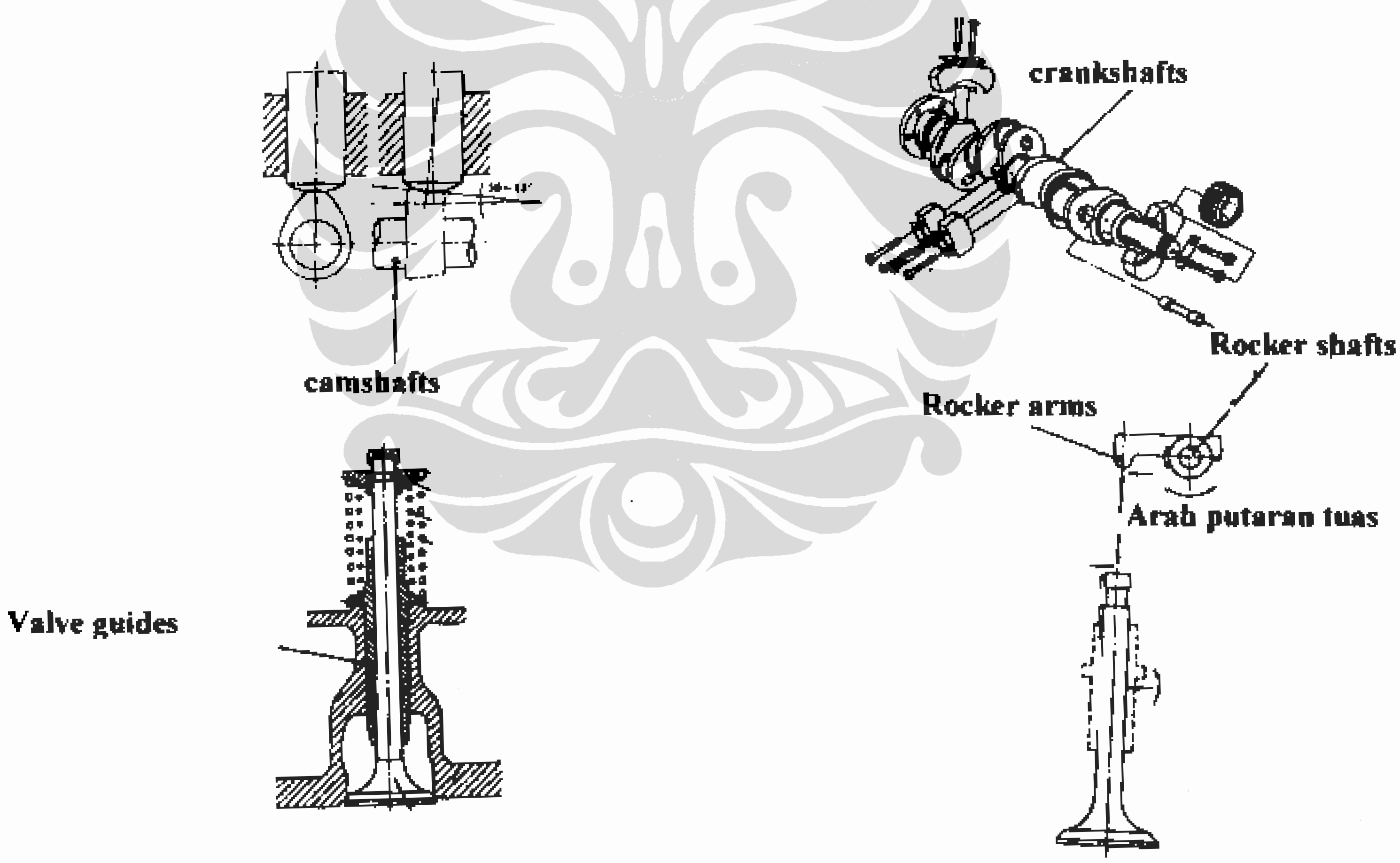

Figure 1: Automotive components treated by ferritic nitrocarburising.

Nitrocarburising process can be performed in various treating atmospheres, such as gaseous, liquid, and plasma environments. In the case of a gaseous nitrocarburising, the use of fluidised bed furnaces offers economic advantages when treatments are carried out in batch production scale. The furnace was first developed in early 1970's[2] and designed to perform full range of thermochemical surface treatments. The major problem with this type of furnace was reported as the nonuniformity of temperature of the workpiece[3]. Recent improvement in design[4] makes fluidised bed furnaces widely used in heat treatment industry. Since the formation of ε $Fe_{2-3}(N,C)$ phased layer is required and this has been commercially achieved by other gaseous nitrocarburising using sealed quenched furnace (e.g. Nitrotec). In this work therefore, formation of compound layers resulting from fluidised bed nitrocarburising technique are studied in relation with processing parameters.

Experimental Methods

Carbon steel AISI 1040 (~0.4 wt.% carbon) supplied as annealed condition was employed in the present sequence of experiments. This low-grade material is widely used for nitrocarburising since the treatment gives favourable scuffing resistance (adhesive wrear resistance).

The nitrocarburising was performed at a temperature of 570 °C in atmosphere containing NH_3+N_2+LPG gas additions. Treatment time was set at 1 to 5 hours. The fluidised bed furnace used during the course of works was commercial type with size of chamber having ϕ 1 m x 1.5 m dimension. The whole series of treatments carried out during the works[5] are shown in Table 1.

Table 1 Series of experiments

Experiment	Gas composition (vol.%)	Treatment time
A	50%NH_3 + 1% CO_2 + 49% N_2	1, 2, 3, 4 hours
B	50%NH_3 + 2% CO_2 + 49% N_2	3, 4 hours
C	50%NH_3 + 3% CO_2 + 49% N_2	3, 4 hours

Microstructural analysis of resulting nitrocarburised layers was made by using both optical and scanning electron microscopy methods. Prior to analysis the specimens were etched with 2% Nital plus special reagent (1cc HCl + 10 cc ethanol plus 990 cc 3% Nital) to reveal compound layer structures. X-ray diffraction analysis was made to corroborate microstructural analysis and, thus confirming phases formed in the compound layers from various treatments. This analysis made use of a Philip PW 1050 X-ray difractometer with Cr Kα and a scanning rate of 0.75° per minute. Based on these two analyses the characteristics of nitrocarburised layers were then observed.

Results and Discussions

Compound layer structures developed by the present fluidised bed nitrocarburising treatment were essentially composed of ε $Fe_{2-3}(N,C)$, and γ' $Fe_4(N,C)$. Figure 2 shows a cross sectional micrograph of nitrocarburised layers indicating the presence of a compound layer and a diffusional zone.

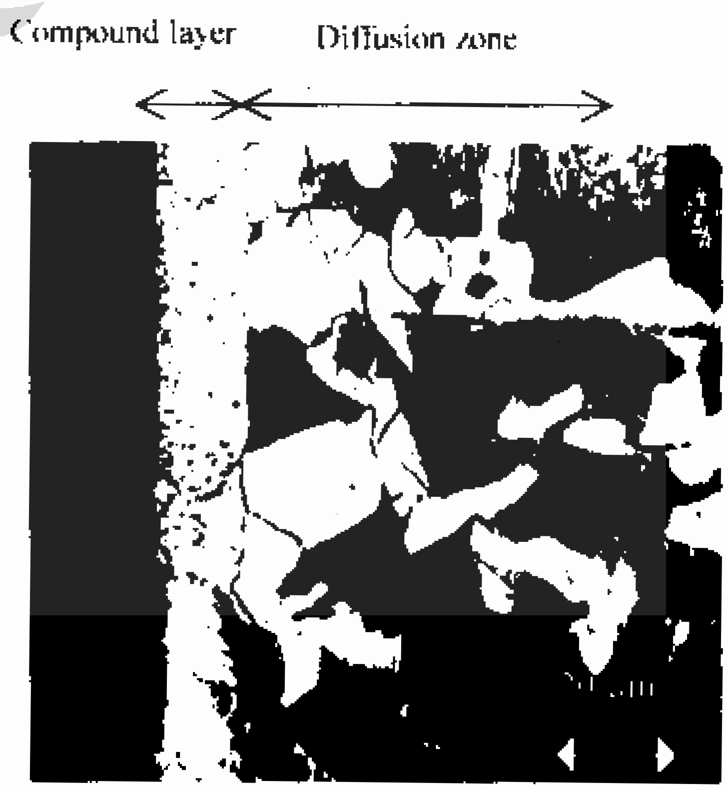

Figure 2: Crossectional picture of a nitrocarburised surface of AISI 1040 steel.

At a fixed gas composition, an increase in treatment time from 1 to 4 hours stabilised the ε phase and at the same time also increased the compound layer thickness. X-Ray diffraction graphs showing stabilisation of ε phase by the increase in treatment time are given in Figure 3.

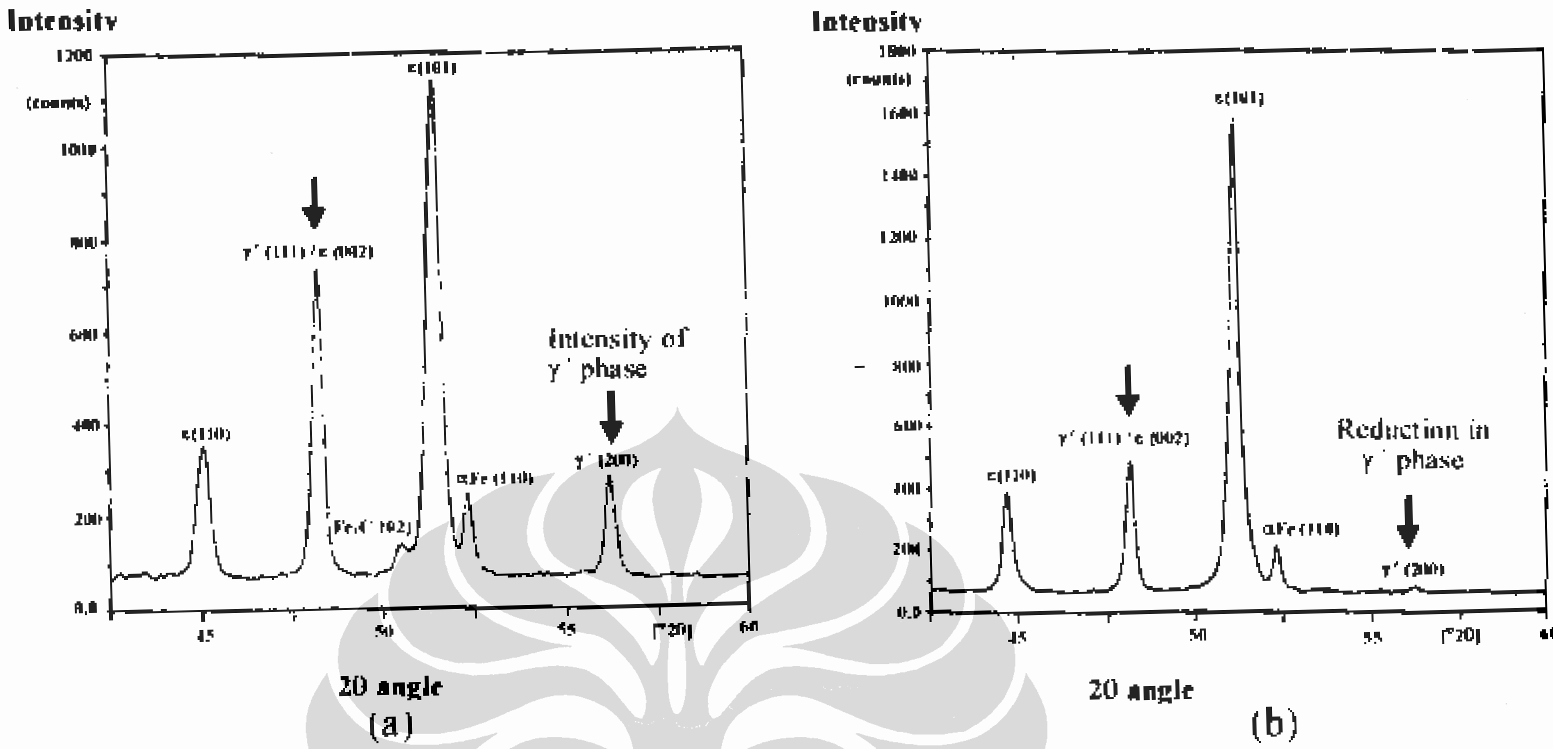

Figure 3 : X-ray diffraction patterns of nitrocarburised specimens; (a) 2 hour treatment time, (b) 4 hour treatment time.

The presence of predominantly ε phased or mono ε phased layer is required to improve scuffing resistance of the nitrocarburised surface [6,7]. A picture of compound layer structures consisting of predominantly ε phase from experiment 'A' with 4 hours treatment time is depicted in Figure 4. In the case of compound layer thickness, experiment showed less doubt that growth rate controlled by diffusional transport of nitrogen and carbon follows a parabolic rate law, Figure 5.

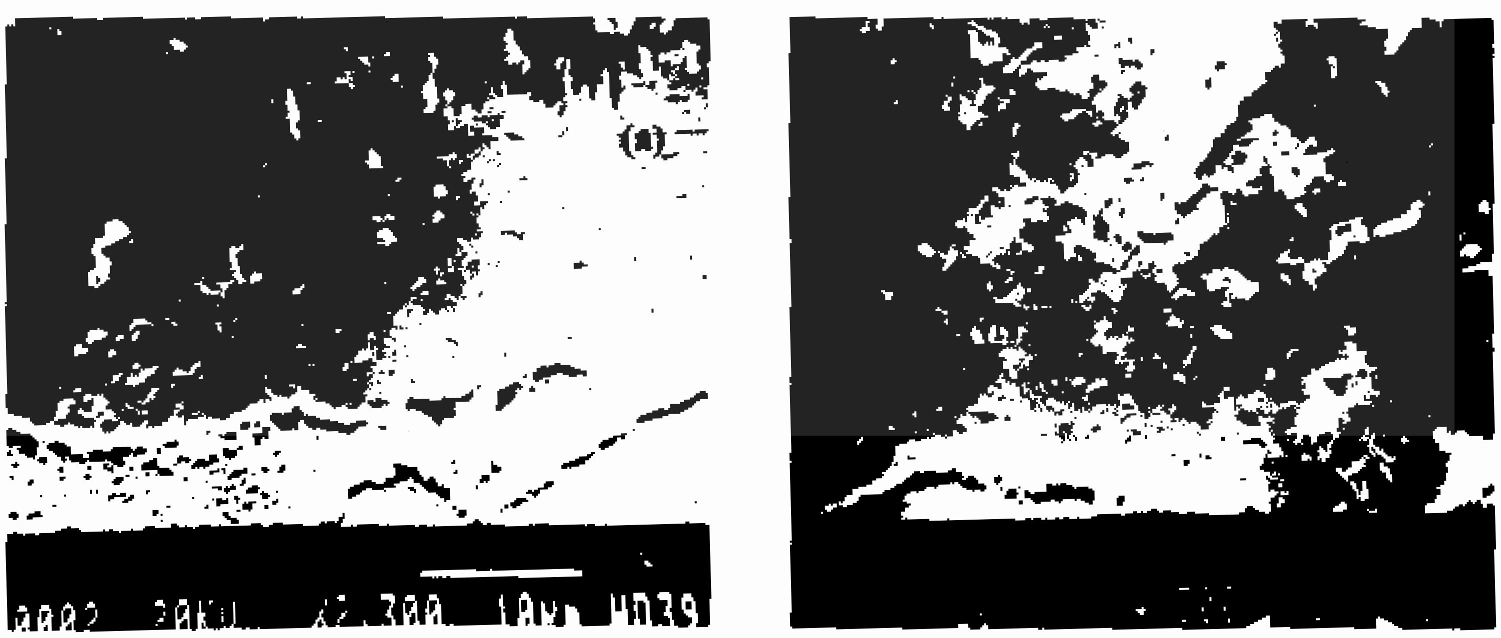

Figure 4 : (a) SEM picture of a predominantly ε phased compound layer. (b) at increased magnification.

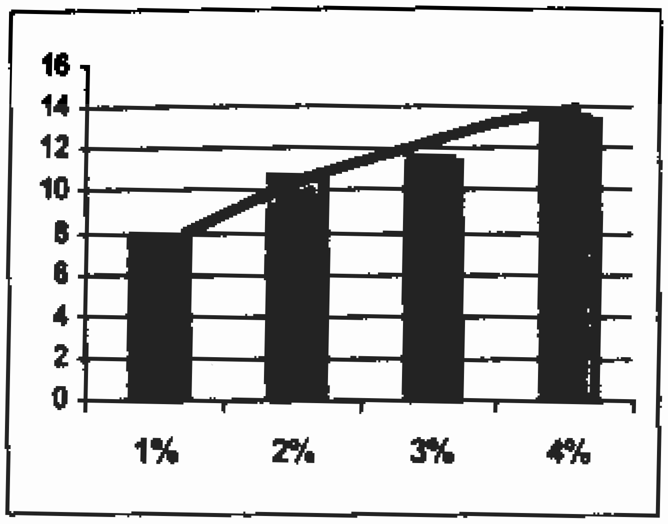

Figure 5 : Growth rate of compound layer formation

Increasing CO_2 gas composition from 1% to 3% produced a thick compound layer with 21 μm in thickness (experiment 'C' 4 hours treatment time). It indicates that the growth of compound layer is not only determined by treatment time but also gas composition. Table 2 shows compound layer thickness resulting from various experiments. Regarding the present use of atmosphere with CO_2 addition, previous work[8] concluded that kinetic of compound layer formation was improved by the presence oxygen in the atmosphere. The formation of ε phase was also stabilised by the presence of oxygen. The mechanism of which oxygen influences this phenomena however is still not well understood [7,8,9]. This is because of lack of information about the influence of oxygen on the stability of various phases in the Fe-N-C system. Accordingly, it is not possible in the present work to quantitatively evaluate the contribution of small amounts of interstitial oxygen to the initiation and development of various phases during nitrocarburising experiments.

Table. 2. Compound layer thickness produced by present experiments (μm)

Experiment	% CO_2	Treatment time			
		1 hour	2 hours	3 hours	4 hours
A	1	8,1μm	10,7 μm	11,6 μm	14,6 μm
B	2	-	-	13,1 μm	14,3 μm
C	3	-	-	18,3 μm	21,4 μm

SEM observations revealed that specimens nitrocarburised with an atmosphere containing 3% CO_2 addition and 4 hours treatment duration (Experiment 'C') exhibited a small amount of porosity in the compound layer located in the outer most of the layer (Figure 6). This tendency, nevertheless, did not occur on specimen nitrocarburised with 1% CO_2 addition. Thus clearly, increasing CO_2 composition will raise nitrogen potential of the atmosphere. As a result, nitrogen in the compound layer also increased.

The formation of porosity in the compound layer can be ascribed due to a denitriding mechanism[7,11]. In this case, when treatment time is prolonged to a such period a recombination of nitrogen occurs due to a metastability of ε phase. This leads to the formation of a N_2 gas molecule with partial pressure as high as 25 Gpa at 570 °C. A stable N_2 gas pore will then grow if the equilibrium pressure of molecular nitrogen exceeds the local pressure or barrier acting on the nucleus. A further mechanism of pore formation as described above is illustrated in Figure 7.

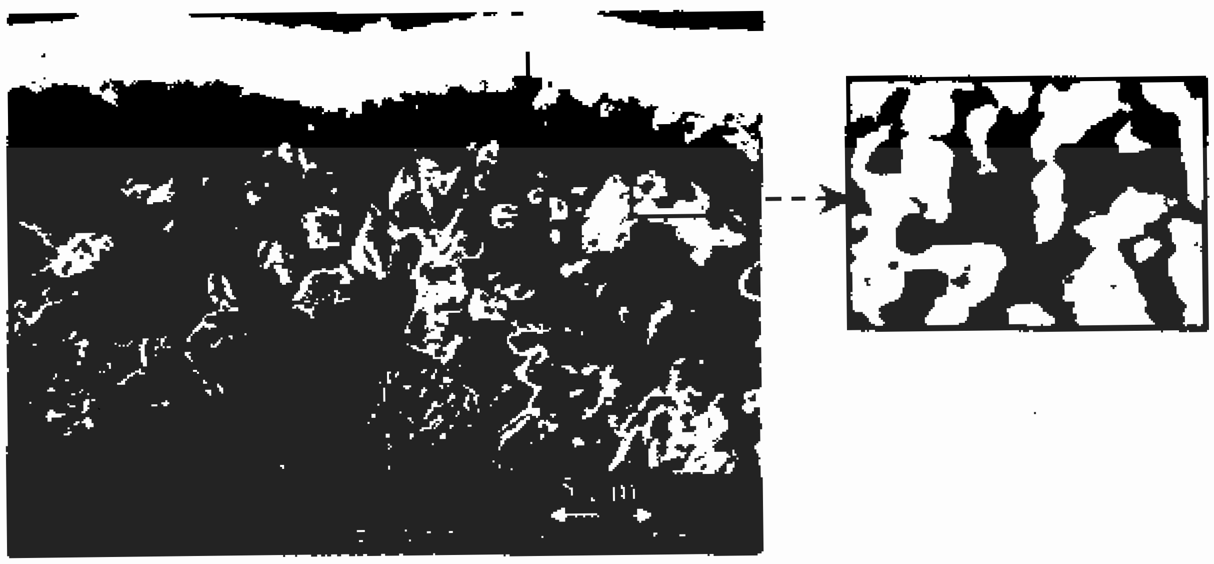

Figure 6 : Pores in the compound layer (indicated by arrows) produced by experiment 'C' with 4 hours duration.

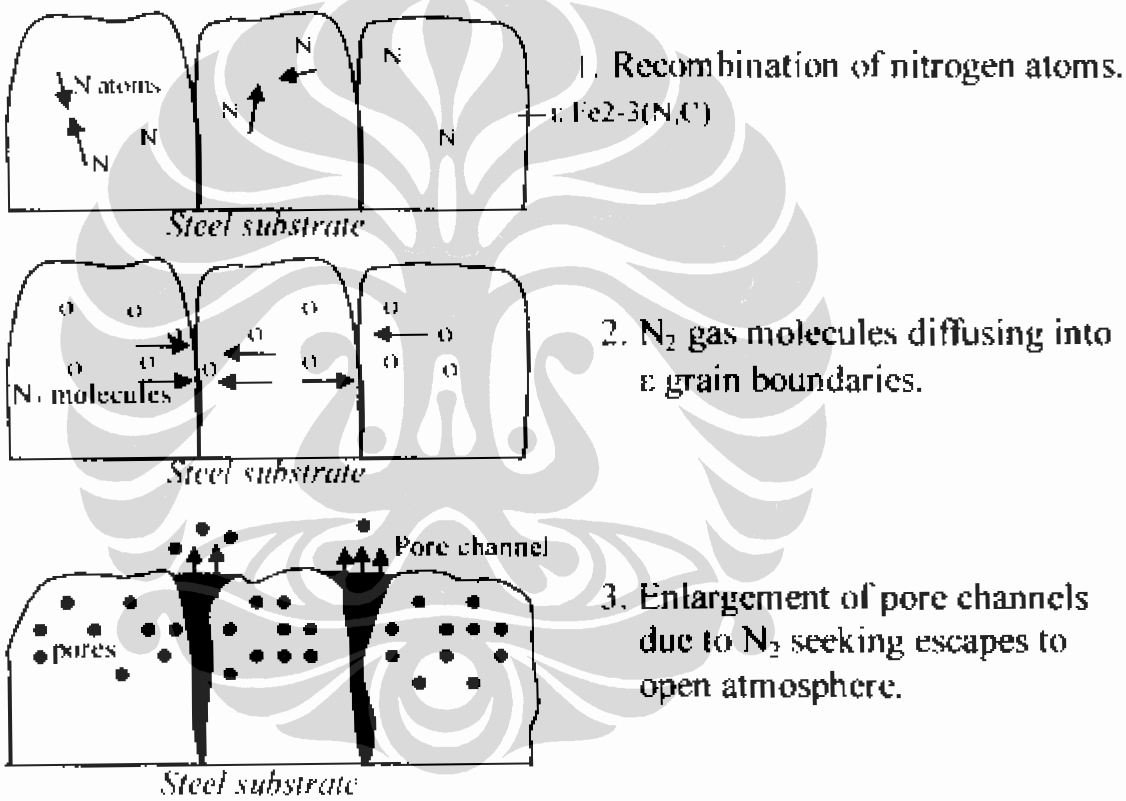

Figure 7 : A model of pore formation.

Conclusions

Based on the present studies some conclusions can be drawn as follows:

- A thick compound layer (up to 22 μm) comprising of predominantly ε phased structure were produced by fluidised bed nitrocarburising using an atmosphere containing CO_2 addition.
- The compound layer thickness was not only influenced by the duration of treatment time as is known elsewhere. The present experiments clearly demonstrated that it was also determined by the CO_2 composition in the atmosphere.
- Formation of porosity in the compound layer was undoubtedly related with a combination between treatment duration and gas composition. Porosity will appear at a shorter treatment time when CO_2 content in the atmosphere increased.
- Because of a high level of porosity was not observed in the present work; nitrocarburising with 4 hours duration and 1%-3% CO_2 gas composition should be presumed enough to produce a superior nitrocarburised

layer (~20 μm in thickness) to combat adhesive wear.

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