PROCESSING CONDITIONS AND HARDNESS DEVELOPMENT IN NITRIDED COMMERCIAL STEELS

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ABSTRACT

The effects of pre-nitriding heat treatments and the influence of alloying e1ements on the development of surface hardness have been investigated using Enl9 and En4lB steels, gas-nitrided at different temperatures and nitriding potentials for 24 to 96 h. The surface hardness is lower at 570^0C than those nitrided at 470^0 and 520^0C. The hardness is reduced with increasing the tempering temperature and this effect is pronounced in En19 steel. The hardness profile is independent of tempering temperatures on nitriding at 520^0C. The profiles are shallower with increasing tempering temperature in Enl9 steel when nitrided at 570^0C. The prior austenitizing temperatures appeared to have no influence on hardness or hardness profile of the nitrided case. The build up of hardness up to a certain depth is established rapidly and remains constant with nitriding time. Below that depth the initial hardness is low and it increases steadily with nitriding time. The combined hardness curves after different treatment times have the appearance of a hysteresis loop.

The results show a constant proportionality of hardness increment to square root of alloy content when nitrided in 80% NH_3 at low temperatures. The hardness increment per percentage of alloying element is sensitive to the choice of base hardness of iron.

The low hardness development at 570^0C is related to reduction in the volume fraction of the fine CrN precipitates due to a coarsening effect and also due to precipitation of coarse nitrides on lath boundaries. This arises since more chromium segregates as $(FeCr)_3$C at high temperatures and in-situ nucleation of CrN occurs at the carbides (mainly at lath boundaries), giving non-hardening coarser CrN. This phenomenon was found to be less effective in aluminium containing steel (En41B). Since aluminium does not segregate to carbide as does chromium in En19 steel, fewer large nitride particles will form and the volume fraction of fine precipitate will be comparatively high in En41B steel.

KEY WORDS: Gas nitriding, Hardness profile, Tempering temperature, Nitriding potential, Hardness loop.

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INTRODUCTION

Nitriding consists of heating steels in a nitrogenous environment usually at a temperature in between 400^0 to 600^0C for a period depending upon the desired depth of the nitrided case. The aim of nitriding is to develop a very hard, wear resistant and corrosion resistant surface and a steep hardness profile of the nitrided case over a tough matrix. Nitriding treatment improves the fatigue properties of engineering components [1]. It is employed as a pretreatment of tool steel substrates for PVD hard coatings which provides hardening and increases load support effect on the substrate [2, 3, 4, 5]. This pretreatment reduces the plastic deformation of the tool substrate which results in eventual coating failure. Sun and Bell [6], Van Stappen et al. [7] and Zeghni et al. [8] found reduced wear on TiN after prenitriding treatment of tool substrates. Prenitriding is reported to increase adhesion [9, 10] and nitrided subsurface improves wear of PVD coatings by more than 1.5 times [11].

In gas nitriding process the total case depth produced depends on the nitriding potential, nitriding temperature, treatment time, prior austenitizing and tempering temperatures, alloying elements present in the steels. For maximum case depth, nitriding is carried out in higher nitriding potential [12, 13, 14, 15, 16] which normally forms an unwanted surface white layer consisting of γ'-Fe_4N alone or a mixture of γ'-Fe_4N and ε-Fe_3N.

The hardness profile of the nitrided materials reflects the amount of fine scale CrN precipitated. In commercial practice it is also used to measure the effective case depth. The degree of hardening corresponds to the depth of nitriding and ahead of which there forms a carbon rich area as shown in Fig. 1 from literature [16]. The surface hardness and the hardness profile development depend principally on processing conditions but other factors such as alloying element concentration in the metal, the ease of nucleation of the nitride precipitate, the strength of the solute-nitrogen interaction and prior tempering treatment time and temperature also influence them. It is reported [15] that plateau of constant hardness and a steep profile gradient can occur in high chromium steels. This plateau of constant hardness and with a steep hardness profile progressively disappears with decreasing alloy content and higher nitriding temperatures. Hodgson and Baron [17] have also shown that by raising the nitriding temperature and carbon content of En40 type steel the hardness profile becomes shallower. However, the effects of nitriding conditions and pre-nitriding treatments on the development of surface hardness of commercial steels have not been studied thoroughly. This paper describes the influences of processing conditions and pre-nitriding tempering treatments on the hardness development of nitrided En19 and En41B steels.

EXPERIMENTAL TECHNIQUE

The chemical compositions of the commercial grade En19 and En41B steels used for this investigation are listed in Table 1. Quenched (900^0C) and tempered (600^0C) specimens of approximate dimensions 4x10xl5 mm were thoroughly electro polished and etched in nital. These were then nitrided in controlled ammonia-hydrogen atmospheres in a horizontal alumina tube furnace. Nitriding was carried out at 470^0, 520^0 and 570^0C in 10%, 30% and 80% NH_3 for 24, 48 and. 96 h.

The effects of prior austenitizing temperature and tempering temperature on hardness of the nitrided steel were studied on En19 steel only. In tempering temperature experiments the specimens were oil quenched from 900^0C and tempered at 500^0, 600^0, 650^0 and 700^0C for I h and these were then nitrided along with as quenched specimens at 520^0 and 570^0C for 48 h in 30% NH_3. In austenitizing temperature experiments, the specimens were austenitisized at 900^0, 1000^0, $1l00^0$, 1200^0 and 1300^0C for 0.5h and then oil quenched. Finally these specimens were tempered at 600 C for 1 h and then nitrided at 520^0C for 48 h in 80% NH_3.

After nitriding, the specimens were plated with nickel and copper in order to preserve the edge of the specimens which tends to break up during polishing of the mounted cross-sections. 2 mm was then ground off from the edge of the plated samples, which were then mounted and polished by standard metallographic techniques. Microhardness measurements were made on specimens in polished and unetched conditions, using a 50g load and a microhardness attachment to a Reichert Projection Microscope.

RESULTS

Effects of Temperature and Nitriding Potential on Hardness Development

Effect on En19
The hardness exhibited after nitriding at different nitriding potentials and times at 470^0, 520^0 and 570^0C are shown in Fig. 2 to Fig. 4. These figures reveal that when nitrided in 10% NH_3 the surface hardness is very low and the hardness profile is very shallow; the hardness increases from 400 Hv at 470^0C to about 460 Hv at 570^0C. This low hardness at low ammonia contents indicates a tendency to weak interaction between chromium and nitrogen.

The maximum hardness has increased with increase of NH_3 content in all three temperatures, see Figs. 2-4. The highest hardness of 750 Hv is achieved after nitriding at 470^0C in 80 NH_3 and it reduced to 600 Hv with increasing temperature to 570^0C (Fig. 4).

The hardness profile becomes slightly steeper with increased ammonia content at 470^0C, see Fig. 2. As the nitriding temperature increases the surface hardness is found to decrease slowly and the profile becomes shallower. With increase of nitriding time a very small increase in hardness is observed. The result for nitriding for 24 h at 520^0C in 30% NH_3 (Fig. 3) must be regarded as anomalous.

Effect on En41B
The hardness build up in this steel is shown in Figs. 5 to Fig. 9. At all nitriding temperatures the maximum surface hardness is found to increase with increase of NH_3 content in the gas mixture. At 470^0C in 10% NH_3 the maximum hardness is very low, about 530 Hv when nitrided up to 48 h (see Fig.5). This low hardness at 10% ammonia content indicates a weak interaction between A1 and N to form the nitride. This low hardness is also observed when nitrided at 520^0 and 570^0C, see Fig. 7 and Fig. 8 respectively.

With higher treatment temperatures the maximum surface hardness is found to decreases i.e. 1250, 1150 and 1100 Hv at 470^0, 520^0 and 570^0C respectively in 80% NH_3. But when nitrided in 30% NH_3 the nitriding temperature has little influence on the maximum hardness. But in 10% NH_3 there appears a slight variation in the hardness. The hardness profile is found to be very steep when nitrided at 470^0C (Fig. 6) and this steepness is also observed at 520^0C when nitrided for 24 h (Fig. 7). The profile is found to be relatively shallower at 570^0C, see Figs. 8 & 9.

The maximum surface hardness is found to increase with treatment time when nitrided at 470^0C, but at 520^0 and 570^0C it is found to be almost constant. The highest hardness observed in this steel is 1250 Hv, produced at 470^0C in 80% NH_3 and nitrided for 96 h. The hardness profile at 470^0C remains unchanged with time but it becomes shallower at higher nitriding temperature.

At 520^0 and 570^0C the maximum surface hardness is found to build up rapidly to a certain depth and then remains constant with time. Below this depth the maximum hardness is initially low at a constant value independent of ammonia content at a particular temperature, but with time the hardness increases steadily to form a "loop" This behaviour is observed in all ammonia contents at 570^0C, see Fig.8 and Fig. 9, and 30% and 80% NH_3 at 520^0C, see Fig. 7. The constant-hardness level at a certain depth is found to decrease at higher nitriding temperatures, i.e. it is 840 and 660 Hv at 520^0 and 570^0C respectively. This type of profile is also observed in En40B steel nitrided at 570^0C in 80% NH_3 [18] but not at all in En19 steel in this investigation.

Effect of Tempering Temperature

The effect of prior tempering temperatures on hardness profile was investigated by nitriding En19 steel at 520 and 570^0C in 30% NH_3. The results are plotted in terms of hardness against case depth in Figs. 10 & 11 and the results indicate that hardness profiles get shallower at higher tempering temperatures. The figures show that the maximum surface hardness of 760 Hv attained in the as-quenched specimen nitrided at 520^0C and it decreased progressively with increase of tempering temperature. When nitrided at 570^0C (Fig. 11) the maximum surface hardness is found to be 630 Hv with the as-quenched specimen and it decreases progressively with tempering temperature similar to what observed by nitriding at 520^0C in Fig. 10. The results in Fig. 10 indicate, however, that tempering temperature has little influence on hardness profile; however, it becomes shallower when subsequently nitrided at higher temperature (570^0C). It should be noted that the core hardness decreases substantially with increasing tempering temperature.

Investigation with En40B (3% chromium) steel it was found that the surface hardness decreases with increasing tempering temperature, the hardness profile is independent of tempering temperatures on nitriding at 520^0C but shallower with increasing tempering temperature when nitrided at 570^0C [18]. Barker and Smith [19] came to a similar conclusion, i.e. the nitrided surface hardness of En19 steel progressively decreases with increasing tempering temperature, the total decrease being less in higher chromium steels. They also stated, in agreement with Hodgson and Baron [17] that in general, the nitrided surface hardness will be largely dependent on the initial hardness of the steels regard1ess of how it was achieved, i.e. by what combination of time and temperature. The decreased surface hardness with increasing tempering temperatures in this investigation confirms the findings of previous workers [17, 20, 21].

Hodgson et al [17] concluded that decreased surface hardnesses at higher tempering temperatures are pronounced with steels of over 0.3% C.

Effect of Austenitizing Temperature

The influence of austenitizing temperature on hardness is illustrated in Fig. 12 which clearly shows that austenitizing temperature does not have any effect on the maximum surface hardness or hardness profile of the nitrided case. Similar results are reported earlier with En40B steel [18].

DISCUSSION

Hardness Increment

The high hardness of the nitrided layer is attributed to precipitation of a very fine dispersion of CrN and the maximum surface hardness increases with increase in chromium content, i.e. increase of CrN precipitate density at the surface. The maximum hardness increment (above that of the fine-grained iron, not the tempered martensite since the matrix of the nitrided layer is free of carbide) should, therefore, be dependent on the alloy content. The maximum surface hardness of these two nitrided steels and of nitrided En40B steel from literature [18] at different nitriding conditions and also the increase in hardness taking a nominal value of 200 Hv for fine-grained ferrite are shown in Table 2. The hardnesses of some nitrided alloys from Lightfoot's [13] work are shown in Table 3 for comparison. It can be seen from the Table 2 that apart from the steels nitrided at 570^0C the increase in hardness is proportional to the square root of the volume fraction of chromium nitride, i.e., $\Delta H \propto \sqrt{X}$, where X = substitutional alloy content. The results show a constant proportionality of hardness increment to square root of alloy content when nitrided at 470^0 and 520^0C in 80% NH_3. The hardness increment per percentage of alloying element is sensitive to the choice of base hardness of iron.

The present results also show that the increment of hardness per percentage of alloy content increases with increase of ammonia content and decreases with increasing the nitriding temperature. The low hardness at 570^0C occurs because of a reduction in the volume fraction of the hardening fine CrN precipitates due to a coarsening effect and also due to precipitation of coarse nitrides on lath boundaries. This arises since more chromium segregates as $(FeCr)_3C$ at high temperatures and in-situ nucleation of CrN occurs at the carbides (mainly at lath boundaries), giving non-hardening coarser CrN. This phenomenon was found to be less effective in aluminum containing steel (En41B). Since aluminum does not segregate to carbide as does chromium in En19 steel, fewer large nitride particles will form and the volume fraction of fine precipitate will be comparatively high in En41B steel.

The effect of low ammonia content on the proportionality constant can be attributed to incomplete precipitation at low fluxes of nitrogen. The total hardness development in nitrided steel depends on many factors and they are mainly; prior tempering temperature, nitriding temperature, concentration of alloying element and carbon content in steels.

Tempering Temperature

It has been shown in Figs. 10 & 11 that the maximum surface hardness of En 19 steel decreases with increase of tempering temperature. The hardness profile also gets shallower at higher tempering temperature. In an earlier investigation with En40B steel a similar result, i.e., shallow hardness profile at increasing tempering temperature, was observed when nitrided at 570^0C [18]. However, the effects are more pronounced in this low chromium En 19 steel compared to that with EN40B steel [18]. The main reason for lower hardness at higher tempering temperature is that at high temperatures larger carbide particles (containing high chromium) are precipitated and on nitriding these carbides are destabilized as chromium is transferred from the carbide to the nitride which begins to nucleate at the surface of the carbide. Dissolution of this carbide particle takes place slowly and as a result the nitride particle gets bigger and contributes little to higher hardness.

When the alloy addition is low and the carbon content is high, a large proportion of chromium will precipitate as carbide on tempering, and little will remain in solution. So on nitriding the amount of hardening by fine nitride particle precipitation will be less. When nitrided at higher temperature the coarsening effect is cumulative, and hence the hardness profile becomes very shallow at 570^0C. In high chromium and low carbon steel the effect is not expected to be so severe since large amounts of chromium remain in solution after tempering and the proportion of fine nitride particles will be higher.

Nitriding Temperature

Increasing the temperature of nitriding leads to an increased depth of hardening but also leads to a reduced surface hardness and a shallower hardness profile, particularly at 570^0C (Figs. 4 & 9). This arises since during nitriding the iron chromium carbide particles produced during tempering dissolve, displacing carbon ahead of the nitrided front where it reprecipitates to form carburised layer [22]. It has been shown [23] that in the carburised layer the carbide particles grow very large compared to those in the core. Increasing the nitriding temperature will further increase the size of these carbide particles and hence their chromium content. On the subsequent advance of the nitrided layer these coarser carbides, which take a longer time to dissolves, transform to non-hardening large nitride particles and so the build up of hardness is much slower.

The formation of a hardness "loop" in En41B steel (Figs. 7-9), not in En19, can be explained in terms of alloy content and the coarsening effect of the carbides in the carburised layer. Since the carbon content in the carburised layer increases with increasing treatment time, in the initial stage of nitriding the increase of carbide concentration, and the concentration of chromium in the carbides will not be so great. Hence, up to a certain depth, the hardness builds up rapidly to its maximum value. With further increase in nitriding time the carbide concentration in the carburised layer increases substantially and with it the chromium content in the carbides, thus depleting the matrix of chromium. The coarsening of carbides and the depletion of chromium in the matrix occur simultaneously. The maximum possible hardness below this depth therefore decreases, due to the increased amount of coarse nitride, and the build up of hardness is slow because more chromium must be supplied from dissolving carbides. If, after a certain times, the amount of chromium contained in the carbides of the

carburised layer becomes constant then the maximum achievable hardness will also be constant (see Fig. 13).

Referring to Fig. 13, if H_1 is the maximum hardness at the surface and H_2 is the constant maximum hardness below a critical depth then ΔH, the total hardness drop due to the coarsening effect, is given by: $\Delta H = H_1 - H_2$. ΔH increases with increase of the nitriding potential and temperature, as has been shown in Figs. 7-9.

Now: (a) when the alloy content is high, the hardness below the critical depth will build up rapidly to the constant level H_2 because of precipitation of a large volume fraction of fine nitrides from the chromium or aluminum in solution, so a hardness "loop." occurs, see Fig. 13a.

(b) When the carbon content is high and the alloy content is low, the hardness will be developed mainly by the chromium released from dissolving carbides and it will build up very slowly towards the constant hardness which leads to shallow hardness profile. In this circumstance no "loop" will occurs (Fig. 13b) as has been seen in Figs. 2-4 for En19 steel.

(c) When the carbon content is low but the alloy content is high, it will take a longer time or a high temperature to build up the hardness "loop", i.e., to achieve a constant carbide concentration and hence chromium concentration in the carburised layer. If the alloy content is very high, there may not form any hardness "loop".

(d) When the carbon content is high and alloy content is high as well, the hardness "loop" will form at shorter nitriding times and lower nitriding temperatures since the constant carbide and chromium concentrations in the carburised layer will be achieved in a shorter time.

At lower nitriding potentials and nitriding temperatures, the hardness build up will be slow because of the low reactivity to form nitrides. It will take a long time to reach those constant concentrations of carbide and chromium in the carburised layer, and secondary coarsening of CrN may take place during this long period. However, if it does reach that critical level the hardness "loop" should occur.

Effect of Alloy Content and Nitriding Potential

The lower hardness build up in steels containing less chromium when nitrided in 10% NH_3 at 470^0C (see Fig. 2) suggests that at low fluxes of nitrogen or chromium, the reaction to form CrN is limiting and the characteristics of weak interaction are obtained. In En 41B steel at a low flux of nitrogen (Fig. 5) very low hardness is achieved in the nitrided layer and the implication is that no AlN is formed under these nitriding conditions. At higher nitrogen activity, the reaction to form alloy nitride is rapid and hence a higher surface hardness develops due to the formation of finer particles, see Figs.7-9.

Effect of Carbon Content

The effect of increased carbon in En19 on the development of hardness is similar to that of high nitriding temperature as has been discussed previously. The hardness

profiles in Figs. 2-4 show a diffuse interface between the case and the core for En19 steel and it is less so with En41B steel, see Figs. 5-9. The diffuse interface and slow build up of the hardness are proposed to be an effect of the carburised zone ahead of the nitrided layer where coarse carbides containing high chromium are formed. On subsequent advance of the nitriding front it takes longer to transform these chromium containing carbides to CrN, hence hardness build up in this region is slow and the interface is diffuse. It is apparent from this effect of the carburised layer that it is impossible to have a hardness profile with a sharp interface between the case and the core or between the nitrided layer and the carburised layer.

CONCLUSIONS

a) The hardness development is slow for both the steels with low NH_3 especially when nitrided at 470^0C. The maximum surface hardness of 700 Hv was achieved in En19 steel after nitriding in 80%NH3 but it reduced to 600 Hv when nitrided at 570^0C. The profile is shallow almost in all nitriding conditions.

b) For En41B steel the maximum hardness was found to decrease with higher treatment temperature, i.e., 1250, 1150 and 1100 Hv at 470^0, 520^0 and 570^0C respectively in 80%NH_3. The hardness profile is very steep and it decreases slightly after nitriding at 570^0C.

c) The decrease in surface hardness at higher nitriding temperatures is due to coarsening of nitride particles, especially CrN. The shallower hardness build up is because of coarser CrN precipitation which has actually transformed from large carbide particles in the carburised layer.

d) The surface hardness and hardness profiles of En19 steel are significantly affected by prior tempering temperature particularly when nitrided at 570^0C. The as-quenched specimen produced the highest surface hardness after nitriding at 520^0C and this hardness decreases with increasing the tempering temperature. The precipitation of coarser carbides during high temperature tempering subsequently produce larger nitrides which correspond to low hardness development. The austenitizing temperature does not have any influence on surface hardness and hardness profile of the nitrided steel.

e) The combined hardness curves give appearance of hysteresis loop in En41B steel, not in En19, nitrided at 520^0 and 570^0C in higher nitriding potentials. The formation of such loop is because of a rapid build up of hardness from precipitation of a large volume fraction of fine chromium nitrides from the chromium in solution. When the solutes content is low the hardness build up will be slow because of the slower transformation of the coarse carbides from the carburised layer to large nitrides which then form the major fraction of nitrides. In this case there does not form any hardness loop.

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Table 1. Chemical compositions (in wt%) of the alloys investigated

Alloy	C	Cr	Mo	Ni	V	Al	Si*	Mn*
En19	0.37	1.03	0.02	0.23	0.02	-	0.1-0.35	0.5-0.8
En41B	0.46	1.60	0.30	0.40	-	0.85	0.40	0.60

(S= 0.05% and P = 0.05% in each steels), * Specification only - no analysis obtained.

Table 2. Proportionality constants of maximum hardness of steels, considering the base hardness equal to 200 Hv.

Nitriding temp., ^{0}C	Steel	Alloy content, at%	Nitrided in 10% NH$_3$			Nitrided in 30% NH$_3$			Nitrided in 80% NH$_3$		
			H	ΔH	$\frac{\Delta H}{\sqrt{Cr+Al}}$	H	ΔH	$\frac{\Delta H}{\sqrt{Cr+Al}}$	H	ΔH	$\frac{\Delta H}{\sqrt{Cr+Al}}$
470	En40B		840	640	366	960	760	435	1050	850	487
	En19		420	220	209	630	430	409	750	550	522
	En41B		770	570	307	1025	825	444	1240	1040	560
520	En40B	Cr=3.05	925	725	415	930	730	418	980	780	447
	En19	Cr=1.108	470	270	244	700	500	475	720	520	494
	En41B	Cr+Al=3.45	830	630	339	1080	880	474	1150	950	511
570	En40B		500	300	172	780	580	332	850	650	372
	En19		470	270	244	550	350	333	600	400	380
	En41B		780	580	312	990	790	425	1100	900	484

H= Maximum surface hard., ΔH=Total increase in hardness, Cr+Al= Weight of Cr plus Al in atomic %.

Table 3. Hardness of alloys nitrided in 27.5%NH$_3$ [1].

W/O Cr	W/O C	Heat treatment	Nitrided at 500^{0}C			Nitrided at 575^{0}C		
			H	ΔH	$\frac{\Delta H}{\sqrt{Cr\,wt\%}}$	H	ΔH	$\frac{\Delta H}{\sqrt{Cr\,wt\%}}$
5.6	-	Annealed	1000	850	350	1000	850	350
2.3	-	Annealed	650	500	322	650	500	322
5	0.2	1h 600^{0}C	950	800	347	640	490	213
2	0.2	1h 600^{0}C	640	490	337	440	290	200
5	0.8	19h 700^{0}C	650	500	217	400	250	108

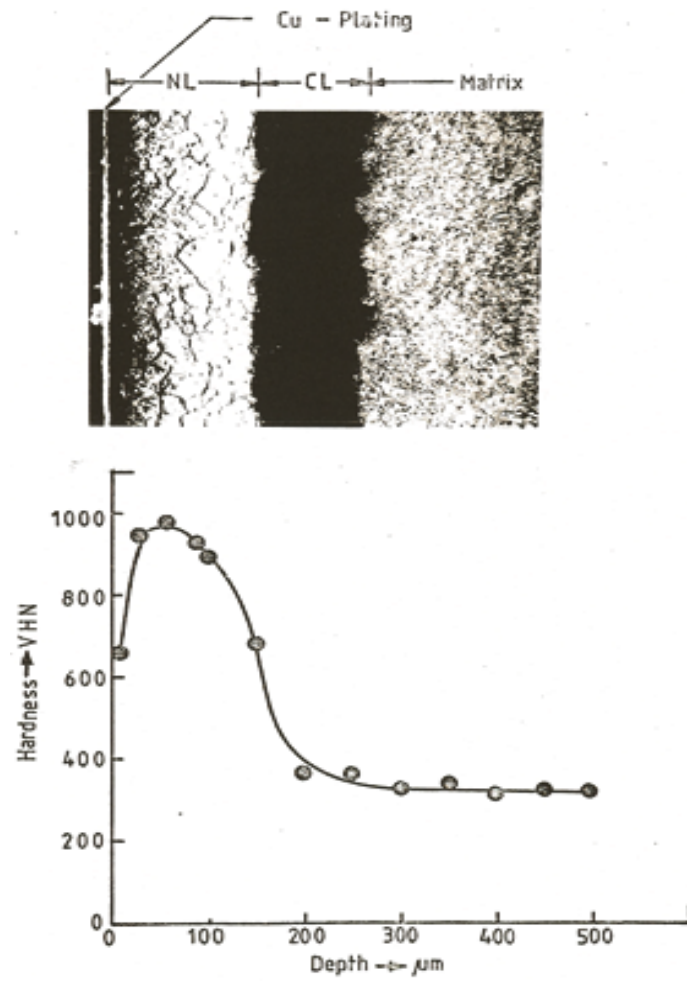H= Maximum surface hardness, ΔH= total increase in hardness.

Fig. 1. (top) Nitrided case with a carbon rich zone ahead: (bottom) hardness profile.
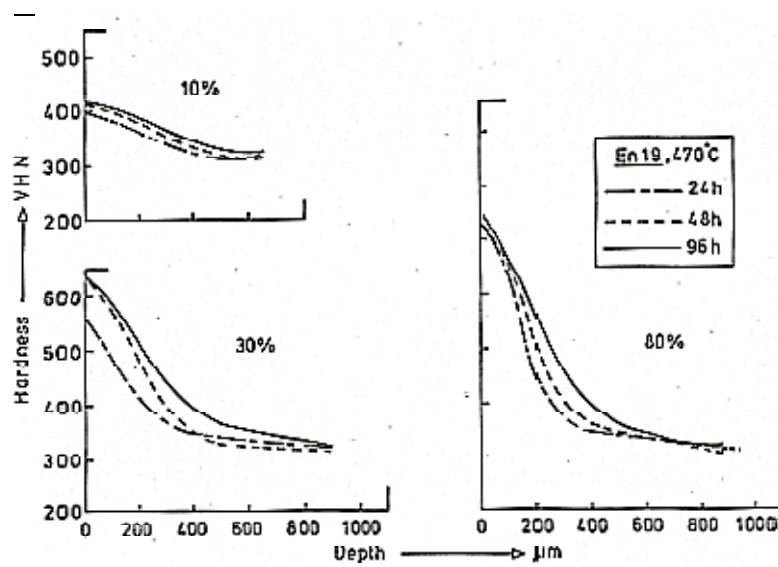

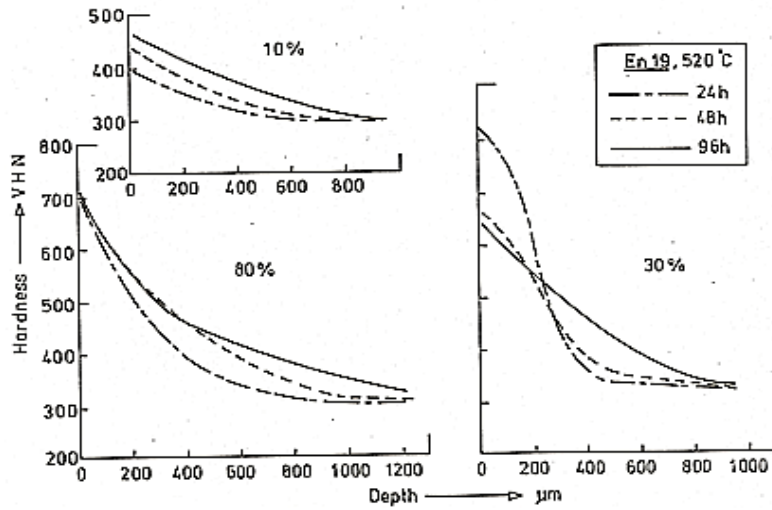**Fig.2**. Hardness profile of En19 steel nitrided at 470^{0}C.

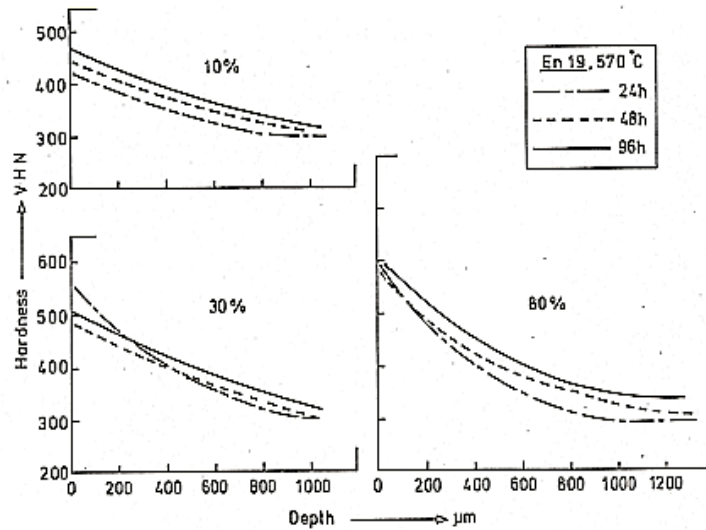**Fig. 3**. Hardness profile of En19 steel nitrided at 420^{0}C.

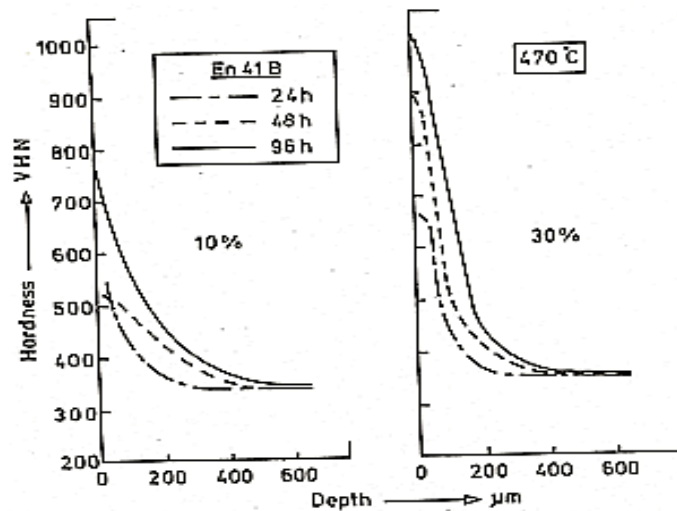**Fig. 4**. Hardness profile of En19 steel nitrided at 570^{0}C.

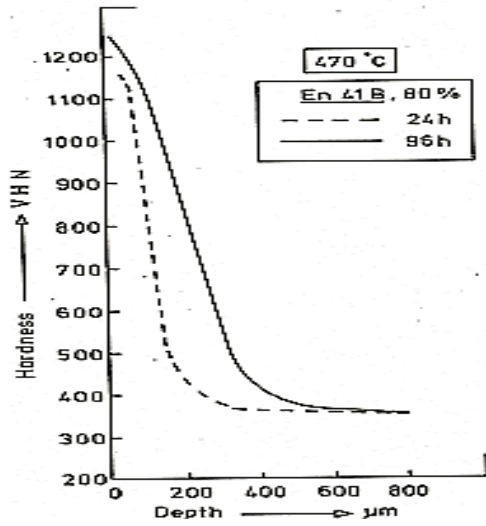**Fig. 5**. Steep hardness profiles of En41B nitrided at 470^{0}C in 10% and 30% NH$_3$.

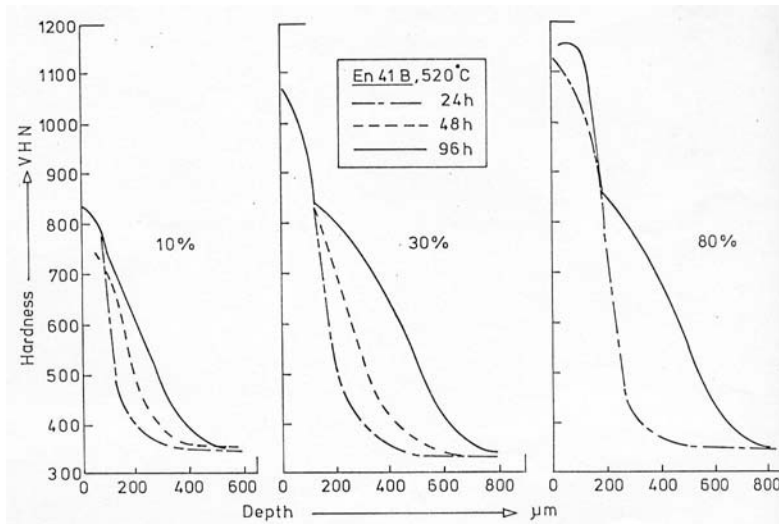**Fig. 6**. Steep hardness profiles of En41B nitrided at 470^0C in 80% NH_3.

Fig. 7. Steep hardness profiles of En41B nitrided at 520^0C.
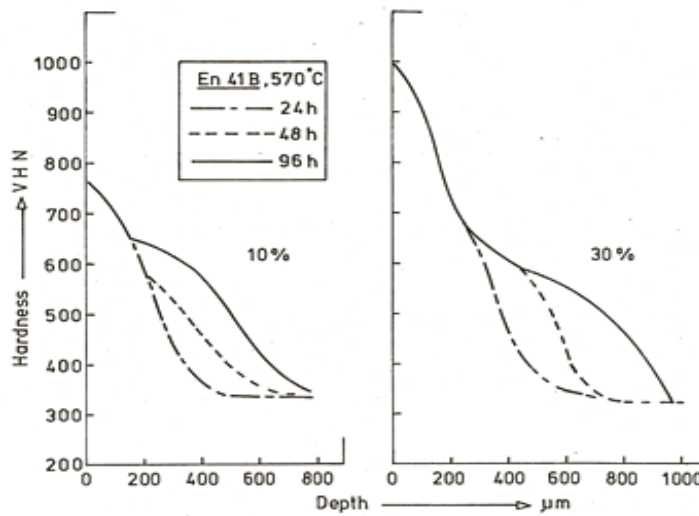
Fig. 8. Hardness profiles of En41B nitrided at 570^0C in 10% and 30% NH_3.
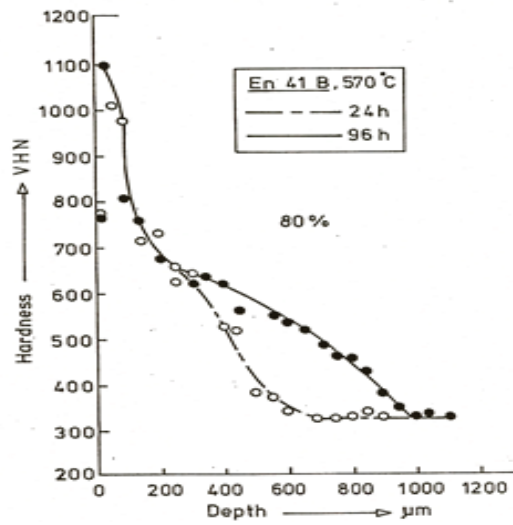
Fig. 9. Hardness profiles of En41B nitrided at 470^0C in 80% NH$_3$.
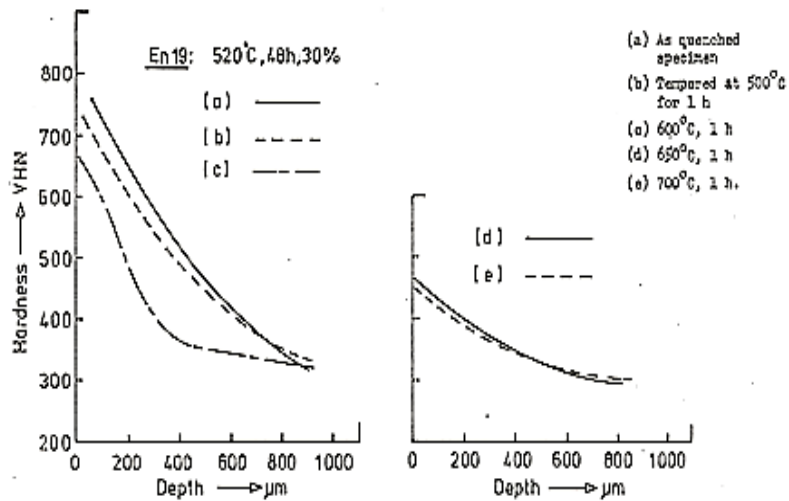
Fig.10. Tempering temperatures and hardness profiles of En19 steel nitrided at 520^0C.
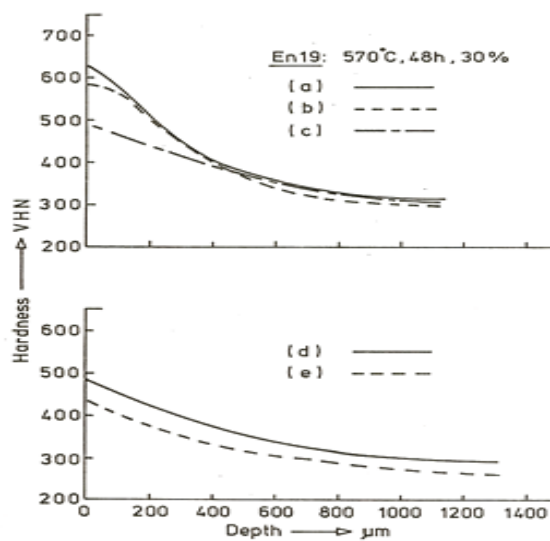

Fig. 11. Tempering temperature and hardness profiles of En19 steel nitrided at 570^0C.
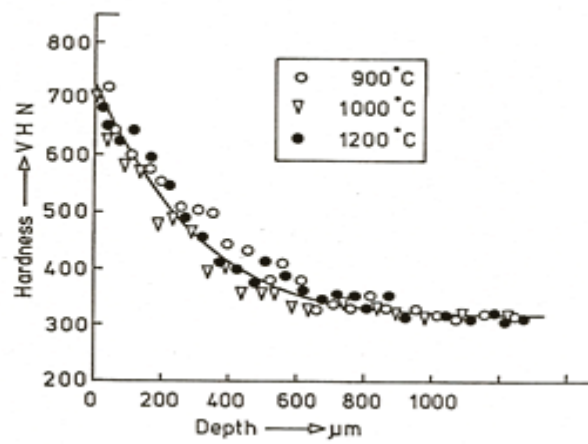
Fig. 12. Austenitizing temperatures and hardness profile of En19 steel nitrided at 520^{0}C.
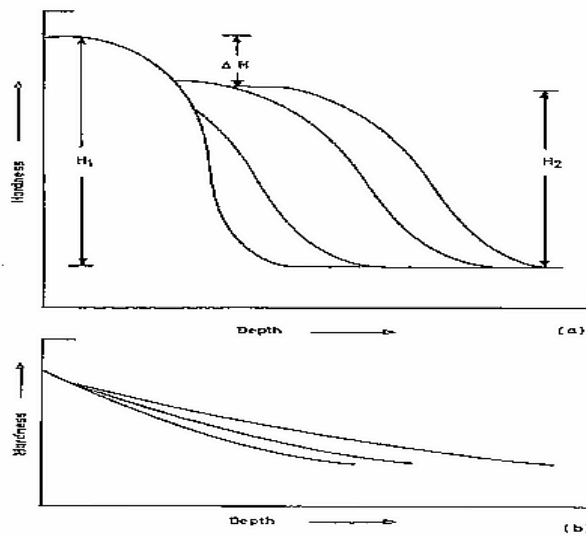

Fig.13. Schematic diagram showing the proposed hardness development and occurrence of hardness loops at successive depths of the nitrided layer (a) high alloy and (b) low alloy.