FACTORS INFLUENCING THE FORMATION OF FLAKES IN STEEL

By

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1. INTRODUCTION

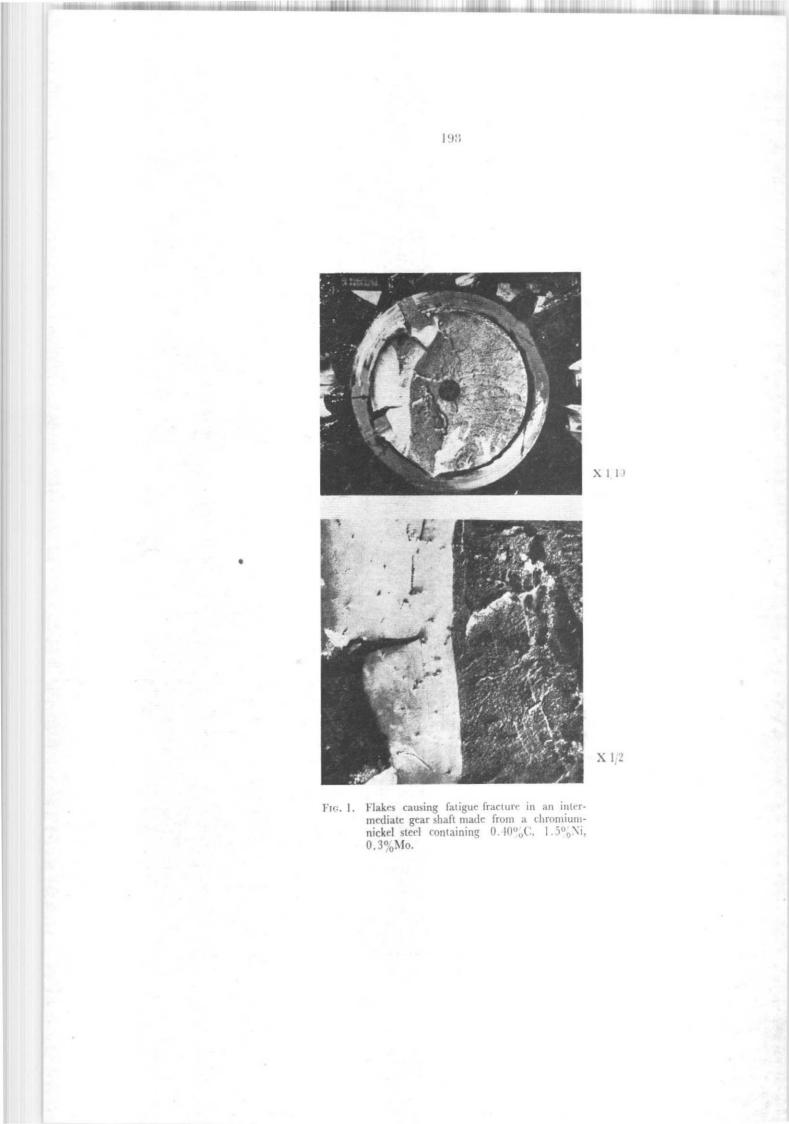
Flakes also called shatter cracks, chrome checks, fish eyes, snowflakes or hairline cracks are internal fissures, which are found to be the frequent causes of failures in service particularly in heavy engineering parts such as axles, crankshafts, rotors, posts of heavy shears or presses and many other instances. Due to their very sharp borders these flakes initiate fatigue fractures. An example of this is given in Fig. 1 which shows the fracture of a large intermediate Sheet Mill shaft broken with a poor service life of only three months. The fracture has extended near the two keyways, but it can be clearly recognised that the fracture started away from the keyways and at some of the flakes distributed in the fracture surface as indicated by the arrows.

Intensive research work has been carried out during the last twenty years to clarify the conditions and causes of the formation of these dangerous internal fissures. While not desiring to lessen the importance of valuable contributions of many individual investigators by omission the work of only a few groups is mentioned since they have been actively and continuously engaged in this field for a period of years and since they are predominantly responsible for the advanced knowledge available to-day. One of these groups is that at Krupps in Germany which published not less than 8 papers ^{1,2,3,4,5,6,7,8}, on this field and was the first to point out the important role which hydrogen plays in the formation of flakes. Very useful and detailed information has been contributed during the last ten years by members of the Hairline Crack Sub-Committee of the British Iron & Steel Institute, which is preferably connected with the names of Andrew 9,10, 11,12, and Sykes. 13,14 These papers have confirmed that hydrogen is an essential prerequisite for the formation of flakes. Some American papers 15, 16, 17, deal with the problem of limiting the primary source of these fissures, namely the hydrogen content of the liquid steel, during steel making For further literature references attention may be drawn to a recent review.¹

The first author was associated with the investigations of the German group right from the beginning as can be seen from the acknowledgement of the firstly referred paper. He has pursued the further developments with own research work and keen interest in the work of others. Since the published results have become very extensive, it is thought worth while to outline in a summarising review the main principles of up-to-date practical experience and to describe the

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 14) J.D. Hobson & C. Sykes, J. Iron & Steel Inst. 170, II(1952) pp. 118/122.
 15) D.J. Carney, J. Chipman, N.J. Grant, A.I.M.E., Electric Furnace Steel Proceedings, 6(1948) pp. 34/45.
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 17) C.E. Sims. G.A. Moore, D.W. Williams, A.I.M.E. 176(1948) pp. 260/278.
 18) A.B. Chatterjea & B.R. Nijhawan, Jour. Scientific & Industrial Research, 11A(1952) 4, pp. 158/161.



scientific and theoretical considerations in as much as they are useful in explaining the procedures applied for either avoiding flakes or reducing the sensitivity of steel to flaking. It was found essential to recalculate some of the earlier results taking the data of more recent investigations as a base, and this has led to a marked alteration in the interpretation in some cases.

2. CAUSES OF FLAKE FORMATION

The theory²) is generally accepted now that the formation of flakes is caused principally by the pressures developed by hydrogen which is expelled during cooling due to a decrease in solubility. When allowed to build up, these pressures may exceed the chohesion strength of steel thus causing gas evolution in the solidification range and ruptures into flakes at low temperatures. In addition to this main reason there are other contributing factors such as stresses, local concentrations and brittleness.

The pressures developed by hydrogen have been calculated¹⁹) for hydrogen contents of 0.001 to 0.003 % and temperatures of 600 to 1700°C. It was considered desirable to revise these calculations on the basis of the most recent solubility data for hydrogen contents which are normally found to occur in steel and to extend them to lower temperatures at least as low as 200°C where steels are susceptible to crack formation. The solubility of hydrogen at one atmosphere pressure is shown as a function of temperature in Fig. 2 which includes the results of several investigators^{19,20,21,22,23,24,25}. When equilibrium solubility values are plotted in cc of hydrogen per 100 g of steel on a logarithmic scale against 1000 divided by absolute temperature T, a straight line relationship is obtained. This permits a linear extrapolation to derive the solubility values at temperatures below 600°C and to correct the values for the & range. It is interesting to note that the solubilities in the α and γ phases become equal at 260°C which is in the temperature range of crack formation.

The pressures developed are calculated by Sieverts²⁰ equation

$$P = \left(\frac{H}{S}\right)^2$$

where P is in atmospheres, H the hydrogen content and S the hydrogen solubility. The results for hydrogen contents varying from 0.5 to 12 cc per 100 g and for a temperature range of 100 to 1650°C are shown in Fig. 3. In the range from 600 to 1000°C the pressures are found to be higher by 30 to 10 per cent than those computed previously.¹⁹) At 1400°C in the γ range, however, the recalculated values of pressures are lower by 20 per cent. But the pressures increase considerably in the δ range, which range was not considered earlier.¹⁹) Therefore gas evolution during solidification can be expected to occur at about 12.2 cc/100 g for primary crystallisation into 8 structure and at a concentration lower than 17.7 cc as can be derived from the calculations for solidification into γ structure. As a further result of the revised calculations, a hydrogen content of 0.5 cc/100 g, which is believed¹³) to be the lowest concentration observed in billets, does not give rise to pressures exceeding the tensile strength of steel at low temperatures. For instance, the the pressure developed by this concentration at 100°C is only about 30 tons/sq. in.

The theory that cooling stresses, caused by the difference in temperature between outside and inside of blooms or billets, are the main reasons of flaking has been discarded. There is no doubt, however, that stresses, whether thermal due to the rate of cooling or produced by transformation or the remnants of mechnical stresses introduced during reduction, do take part in the formation of flakes. The effect of mechanical stresses has been demonstrated⁴) in a very convincing manner by air cooling a forged billet under a tensile load of 12.7 tons/sq. in. which caused an alteration of the crack direction thus creating vertical fissures while longitudinal flakes occurred in a billet of the same size, forged from the same cast and cooled in air without tension. The con-

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 25) H. Liang, M.B. Bever & C.F. Floe, Trans. A.I.M.E. 167(1947) p. 395.

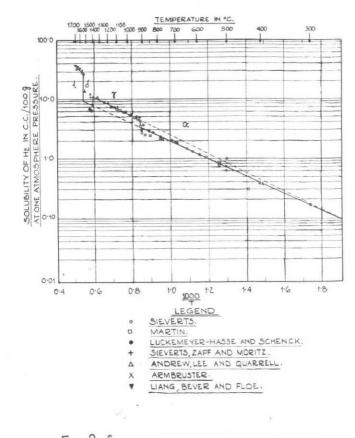
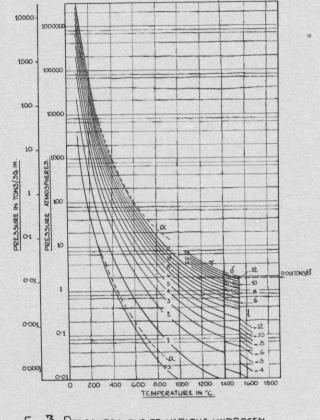
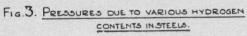


FIG. 2- SOLUBILITY OF HYDROGEN IN IRON AT ONE ATMOSPHERE OF PRESSURE OF HYDROGEN.





tribution of transformation stresses is indicated, for instance, by the observation that nonalloyed steels of different carbon contents when quenched after soaking in hydrogen at 1100°C showed⁴) an increasing number of flakes with increasing carbon content and increasing core hardness. Rapid cooling rates are known to promote the formation of flakes. This is due to the combined detrimental effects of both thermal and transformation stresses. If, however, a steel is soaked in hydrogen under high pressures (1000 at) at temperatures below the transformation range (550°C) and subsequently water quenched or slowly cooled in a separate furnace,⁴) the transformation stresses are eliminated and it might appear from the consequent difference in number of cracks formed that cooling stresses have a considerable influence. It has to be considerd, however, that the smaller number of cracks formed during slow cooling is not solely due to the absence of quenching stresses. It will be shown later that this is caused also by a loss and equalisation of hydrogen which diffuses during the period of slow cooling accelerated by high pressures.

In addition to the hydrogen content and stresses, a few more points are definitely of additional importance as shown clearly by the investigations of the British group. Andrew 9, 10 has observed a hydrogen rich constitutent appearing in the form of white streaks in steels soaked at 1200°C in hydrogen and subsequently water quenched. This microconstituent, which disappears during aging or heating up to 300°C, was stated to be austenitic in nature. This conclu-sion is in conformity with the finding²⁶ that hydrogen causes an increase in hardenability which indicates that it has an austenite stabilising effect. These white streaks of a hydrogen rich constituent are not usually observed in air cooled billets which means that they are normally decomposed during cooling. It does not mean, however, that the local hydrogen concentration as indicated by their occurrence is completely eliminated during the common treatment. The appearance of these streaks on quenching therefore proves, that hydrogen may concentrate not only in cavities and voids but also in structural constituents and that this concentration on a micro-scale can be much higher than average hydrogen contents found by the analysis of samples. Inclusions may also get³ enriched in hydrogen. Thus an hydrogen analysis, which averages over a microscopical area of the sample cannot be expected to express fully the sensitivity to flaking, since existing nonuniformities of microscopical size such as in streaks, voids and inclusions need not be reflected in an overall result.

Since the usual pattern of flakes arranged in longitudinal direction may also occur in slowly cooled blooms, where the stress factor should have very little influence. It has been suggested,¹³ that ductility also plays its part since it is less in vertical direction of a billet. It is true that in small billets, where due to higher reductions the difference in transversal compared to longitudinal properties is more, flakes are mostly found to be orientated in longitudinal direction. In larger blooms, where these differences are less, transversal fissures may be observed more often. This relationship is further supported by the statement,¹³ that structures showing massive banding of ferrite and carbide are more susceptible to flake formation. Further, the nonsensitivity of austenitic steels may be explained to a certain exent by the higher ductility of such steels, since they can stand high local pressures, which are retuced y deformation without rupture due to good cold deformability and high elongation. It will be shown later that it is possible to present an idea about the contributions of these additional factors as compared to that of the main factor namely the pressure of hydrogen coming out from solution

3. EFFECT OF STEEL MAKING PROCESSES.

It is the general experience that the sensitivity of steels to flaking is less in crucible steels, acid open hearth steels and acid high frequency steels whilst basic open hearth steels and basic arc furnace steels particularly the last named are more susceptible¹ to such internal cracks.

From the above comparison may be concluded that the hydrogen content of the furnace atmosphere has little influence since hydrogen absorption from the atmosphere in acid open hearth and in basic open hearth should be equal while the furnace atmosphere in an electric arc furnace contains only very little hydrogen.

²⁶⁾ E. Houdremont & P.A. Heller, Techn. Mitt. Krupp, Forschungsber, 4(1941) pp. 117/126,

Another factor, which is said to be mainly responsible for the above differences in sensitivity, is the, lime addition, since it is much higher in basic processes and the highest in electric arc furnace It has been stated¹³ that in electric are furnace practice, the use of dried limestone, which is less hygroscopic than dried lime, resulted in a liquid steel of a lower hydrogen content than the addition of dry lime having an average moisture content of 0.5 per cent and that the heats made with dried limestone in refining are less sesceptible than heats which are given a single boil and are refined with the use of lime. If calcined lime is left in the normal shop atmosphere, the sur-face layers rapidly absorb upto 10 per cent moisture. Heats made with lime, where no special storage precautions have been used to keep the lime dry in heated furnaces showed¹³ a hydrogen content which was higher by about 2 cc/100g. This may become worse in seasons of high humidity. The different flaking susceptibilities of the different steels as known by experience may be recognised also from the following average figures of hydrogen contents of the liquid steel as determined⁸ at tapping and in the mould.

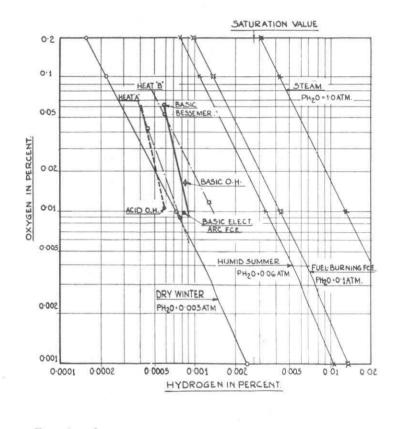
	No of heats. tested	H ² content			
		at tapping		in the mould	
		. %	cc/100g	%	cc/100g
Acid open hearth heats.	10	0.00058	6.5	0.00064	7 · 1
Basic open hearth heats.	19	0.00085	9.4	0.00075	8.3
Basic electric furnace heats.	8	0.00081	9.0	0.00102	11.3
Basic Bessemer heats.	16			. 0.00069	7.6

Obviously, the hydrogen content of basic open hearth and basic steel electric at tapping is higher than that of acid open hearth steel. From tapping to pouring, the hydrogen content in-creases except for the basic open hearth heats, which were tapped non-killed and were killed in the ladle. Sykes¹³ and coworkers, who used a quenched pencil test piece²⁷ for hydrogen determination of liquid steel, which may lose hydrogen particularly during solidification, have generally determined lower figures of hydrogen content than Wentrup⁸ and coworkers who employed a suction method for taking the sample. They confirm, however, that the values of acid open hearth steel are on the average, 4.2 cc/100g (11 heats), i.e. about 0.5 cc/100g lower than those obtained on basic electric steel using dry lime (53 heats) and about 2 cc/100g lower than in normal electric furnace practice using lime without precaution in storage (14 heats). Other investigators ¹⁶, ²⁸, ²⁹ differ with respect to the level of values determined but agree as regards the differences between acid and basic steel. This can be seen from Table 1.

The lower hydrogen content of the blown basic Bessemer steel as compared to the basic open hearth steel is due to the vigorous boil during production and to the higher oxygen content. The oxygen content is a fairly effective influencing factor. This may be seen from Fig. 4 showing the relation between oxygen and hydrogen contents. The relationships calculated¹⁵ for equilibrium conditions at several partial pressures of steam are straight lines when plotted on double logarithmic system. They represent a wide range of different hydrogen availabilities and indicate that the hydrogen content decreases with increasing oxygen content. The results of all the basic processes, when entered in the graph, fall on a line having a slope little different from that of the equilibrium line. This line corresponds to a comparatively low hydrogen availability. The hydrogen availability in the acid open hearth process is obviously much smaller and coincides

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 29) J. Bletin, S. Mischosniky, G. Coin, P. Bastin, Rev. Metall Mem. 48(1951) pp. 471/85.

²⁷⁾ J.E. Wells and K.C. Barraclough, J. Iron & Steel Inst. 155(1949) pp. 27/32.



110. 1.	RELATION BETWEEN OXYGEN AND HYDROGEN
	CONTENT OF LIQUID STEEL ACCORDING TO
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	AND CO-OPERATORS. (1595°C, 2900"F)

with the lowest hydrogen pressures considered. The conditions for the acid Bessemer converter are not yet investigated. They may, however, be expected to lie at higher oxygen contents and on the dotted line for acid processes drawn parallel to that for the basic processes.

This relation is further confirmed by recently reported³⁰ determinations of oxygen and hydrogen contents in the oxidising and reducing periods. If the maximum oxygen content in the former period, and the minimum oxygen content after the slag change are entered in Fig. 4 with the corresponding hydrogen contents, a relation similar to that of the equilibrium lines is found. The difference in the positions of the two heats should consequently be explained by the differences in hydrogen availability. Following this clarification, the observed¹⁶ increase in hydrogen after furnace deoxidation becomes understandable. This increase was stated to be qualitatively comparable to the relative deoxidising power of the ferroalloy added, i.e. there was a slight increase after the manganese addition and a large increase after the silicon addition. This on the whole points out that a highly deoxidised steel bath is more susceptible to pick up hydrogen.

There exists a discrepancy of opinion regarding the effect of aluminium additions. Killing with aluminium caused a loss of hydrogen which was also observed in the pretreatment⁸ of samples. This may be due to a reaction inducing the evolution of hydrogen. If, however, the addition of aluminium is done in the furnace to improve the deoxidation, the affinity for hydrogen absorption may increase.

The difference in the hydrogen contents of acid and basic steels and the high flaking sensitivity of the basic steels has been attributed to the higher water content of the basic slags as expressed by the following data⁸.

	Contained as H ₂	Hydrigen content in Contained as H ₂ O and calcu- lated on H ₂	cc/100g Total	in% Total
acid slags	25 to 60	5 to 40	30 to 100	0.003/0.009
basic slags	10 to 30	30 to 175	40 to 205	0.004/0.019

This shows that at least the hydrogen content as combined into water in basic slags is partly considereably higher than that of the acid slags and of liquid steel and this should increase the availability of hydrogen to the liquid steel baths in the basic processes.

It is an acknowledged fact, that the quantity of hydrogen can be reduced during steel making by maintaining a strong boil the strength of which may be controlled by the rate of carbon drop. This rate is recommended to be between 0.4 to 1.0% C per hour¹⁷ or for acid steel 0.3% C per hour and for basic steel at least 0.5% per hour⁸. The acid operation is stated¹⁷ to be more amenable to such a control. It is worthwhile to note in this connection that the hydrogen and water contents of acid slags decrease⁸ during the boiling period and increase when the carbon drop is over and the heat is kept quiet. Thus the acid slags experience alterations similar to those of the steel bath. Basic slags, however, are not affected similarly and they appear to be more persistant in retaining their higher hydrogen contents. Consequently, the danger of hydrogen absorption is greatest when a heat in a properly deoxidised and in an inactive condition comes across a higher availability of hydrogen as for instance by contact with a basic refining slag or with the residuals of moisture in the runners and ladle. Therefore, it is frequently observed¹⁷ that the hydrogen content jumps up during tapping. Killing in a properly dried ladle may counteract the above effects.

This section may be concluded with some general remarks. It is a repeatedly established experience that in steel plants and in particular in alloys steel plants, where little difficulties with

30) Y. Ishihara and S. Sawa, Proceed. of the First World Metallurgical Congress (1951), pp. 247/255.

flakes were encountered till recently, progressively increasing troubles in this respect were happening. Some reasons for this change become understandable from the above review. The introduction of basic roofs allows to work the heats hotter and to speed up the working periods, but this increases the chances of hydrogen absorption from the basic slags at higher temperatures. Reduction in the proportion of pig iron in the charge to amounts as low as 12 to 15% decreases the opening carbon and shortens the boiling period, thus decreasing the amount of hydrogen removed. The intention to improve the purity of the steel by allowing for better segregation of deoxidation products and to reduce losses in alloying elements requires a fairly complete deoxidation even by partial addition of aluminium into the furnace. This, however, creates conditions which favour the pick up of hydrogen. Considering local conditions, it is obvious that the duplex process with its vigorous boil during blowing in the acid converter and the treatment of the blown metal by a highly oxidising basic slag justifies the expectations of a comparatively low hydrogen content and little sensitivity to flaking. It should be clear, however, that in the triplex process for producing acid steel, where the preblown metal having carbon contents between 2 to 1% C is in contact with basic slags during the period of dephosphorising and the boiling period under the acid slag is shorter than that in direct acid open hearth process, the chances for hydrogen absorption are greater and hence the resulting triplex steels are more sensetive to flaking than the direct acid steels.

4. HYDROGEN ABSORPTION DURING TAPPING AND TEEMING.

The process of tapping a heat into the ladle brings into operation factors which may have opposite influences as regards the alterations of the hydrogen content of the liquid steel. The movement of the steel bath when falling into the ladle and the reactions induced by ladle additions for killing particularly for nonkilled heats may cause a lowering of hydrogen content. On the other hand, the contact of a well deoxidised heat with the residuals of moisture in the runners and ladle refractories may result in hydrogen absorption. It therefore depends on the practice of steel making and the care applied in preparing runners and ladles as the whether the hydrogen content decreases slightly²⁷ (about 0.5 cc/100g) during tapping, or whether the steel becomes spoiled by considerable hydrogen pick up. 17). A newly lined ladle, even after preheating in a normal manner, retains a large amount of moisture in its refractories and a portion of this may remain for the second and possibly also for the third cast. Carbon boil and ladle practice are therefore considered ¹⁷) to be the most important points for producing low hydrogen heats.

During teeming, top poured ingots are found ¹) to be more sensitive to flaking than bottom poured ingots, which is in conformity with the deteriorating effects of the higher teeming temperature and greater teeming speed used in top pouring. Coating of the moulds is an additional factor. The use of noncoated moulds or those which are coated with graphite or soot produces1) steel ingots which are less susceptible to flaking. The important factors in the use of tar and and lacquer for coating the moulds are the temperature of the moulds during coating and thickness of the layer. The increase of hydrogen content due to coating by lacquer was determined⁸) to be about 0.6 cc/100g for acid open hearth steels and about 2.3 cc/100g for basic electric arc steels. This increase which was found by comparison of the hydrogen content of steel samples taken from the mould and the liquid steel from the furnace before tapping was proved ⁸) to be 100 to 300 per cent more for doubly lacquered moulds. The practice of pouring oil on the bottom plates, which produces a deposit of soot on the mould walls during burning off, is permissible if the bottom plates and moulds are sufficiently hot. If, however, a residue of oil remains on the cold bottom plates due to incomplete evaporation, conditions are created which are ideal for hydrogen pick up of the steel poured into moulds prepared in this manner.

Turning to the shape of the ingot section, whether it is round, square or six or twelve sided, no marked difference is to be noticed ¹) as regards the sensitivity of the steel to flaking. The height of the ingot, however, exerts an influence since short and fat ingots favour the escape of hydrogen thus making the steel less susceptible to cracking when compared of the steel cast into tall and thin ingots. Due to a higher ratio surface to weight the core of a short ingot gets enriched in hydrogen to a greater degree and this enrichment may result in the evolution of the gas before the top is solidified and closed. In a tall ingot the top gets closed earlier and thus the hydrogen remains entrapped in the ingot. Further the ferrostatic pressure comes in as a very important additional factor in tall ingots increasing the solubility of hydrogen particularly in the bottom portion. An attempt was made to give an idea about the effects of ingot height, ingot diameter and the degree of solidification on the hydrogen content of the ingot, which by enrichment in the remaining fluid may adjust conditions to cause a loss of hydrogen by evolution. The results of these calculations are compiled in Fig. 5. Firstly the effect due to segregation alone is calculated by the relation.

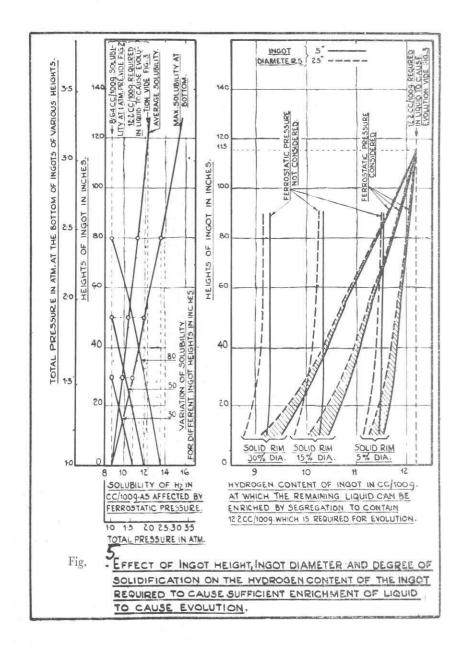
$S=12.2 \times (1-f) + 8.64 \times f$

where S is the hydrogen content of the ingot necessary to cause evolution after a volume fraction f becomes solidified. The values of 12.2 and 8.64 are the hydrogen contents in cc/100g required for evolution from the liquid at 1535°C (taken from Fig 3) and the solubility in § iron at 1535°C at one atmosphere pressure (taken from Fig. 2). When the ferrostatic pressure is considered additionally, the solubility in δ iron varies with the ingot height as shown in Fig 5. at the left. The solubility increases progressively towards the bottom. The alteration of maximum solubility at the bottom and of the average solubility for the range between top and bottom are entered additionally in Fig 5 (left) as dependent on the ingot height. In Fig 5 at the right ingots having two differents diameters of 5'' and 25'' are compared. It is evident, that with increasing propor-tion of solidification the hydrogen content required for evolution decreases considerably which indicates the effect of segregation. The ingot of larger diameter (25'') loses hydrogen by evolution obviously at lower hydrogen contents than the thinner ingot of 5" dia. This difference diminishes with the ingot height. It is clearly understandable now, that a short and fat ingot is more inclined to lose hydrogen during solidification than a tall and thin ingot. If the ferrostatic pressure is taken into account additionally using the averaged value of solubility over the entire solid portion then all the other influences such as those segregation due to the proportion solidified and of ingot diameter decrease with increasing ingot height, revealing the predominant effect of ferrostatic pressure, until at a height of 115" evolution can take place only, if the initial hydrogen content exceed the liquid solubility of 12.2 cc/100g. This explains, why with very high ingots the chance of losing hydrogen due to evolution is small and why traces of hydrogen bubbles are arely observed in the bottom portion because of the increase in solubility caused by ferrostatic pressure. The above calculations should be verified by actual determinations of the hydrogen contents of solidifying ingots. The influence exerted by the closing up of the top part of the solidifying ingot explains the observation² that a smaller tendency to form flakes is found when the solidification of the hot top is slow and delayed. Hence the practice of quenching the ingot top and quick cooling, as recommended ³¹) for increasing the rolling yield, may be expected to increase the flaking sensitivity. Hot top heating, ³²) however, should result in decreased sensitivity. Flakes are not usually observed ²) in small ingots weighing upto 1 Cwt., if the steels are not artificially enriched in hydrogen, since such experimental heats are cast in comparatively short ingots for instance with 6" dia. and 11" height. The steel cast into an ingot of 6" dia. and 55" height and weighing 5 Cwt. is. however, known to show flakes. This is caused by both the greater height and the larger weight. The affinity to form flakes is enhanced progressively with increasing ingot size. Big ingots, weighing 30 tons and over, provided for heavy forgins are therefore cast preferably from acid steels in order to decrease their sensitivity to flaking.

Generally, the steel from the top of an ingot is more susceptible to the formation of flakes than that from the centre or the bottom of the ingot. This indicates an additional effect of segregation. Enrichement of hydrogen at the top has been confirmed ¹⁴) by hydrogen determinations for the cores of large ingots and in increase of hydrogen near the axis was noticeable even after forging. There are considerable variations in the results of the hydrogen analyses on samples taken at short distances apart from one another. This is believed to be caused by segregation into minute cavities and voids. Such cavities get welded during forging and hot rolling, and this forces the entrapped hydrogen to go back into solution and thus accounts for the occurrence of high local concentrations of hydrogen

³¹⁾ G.W. Humes, A.I.M.E. Open Hearth Proceed. 30 (1947) 30th Confer. p. 194.

³²⁾ A.J. Texter & E.F. Kurzinsky, Iron Age 169 (1952) 7, pp. 126/128.



5. THE EFFECT OF HOT WORKING, ANNEALING AND SLOW COOLING.

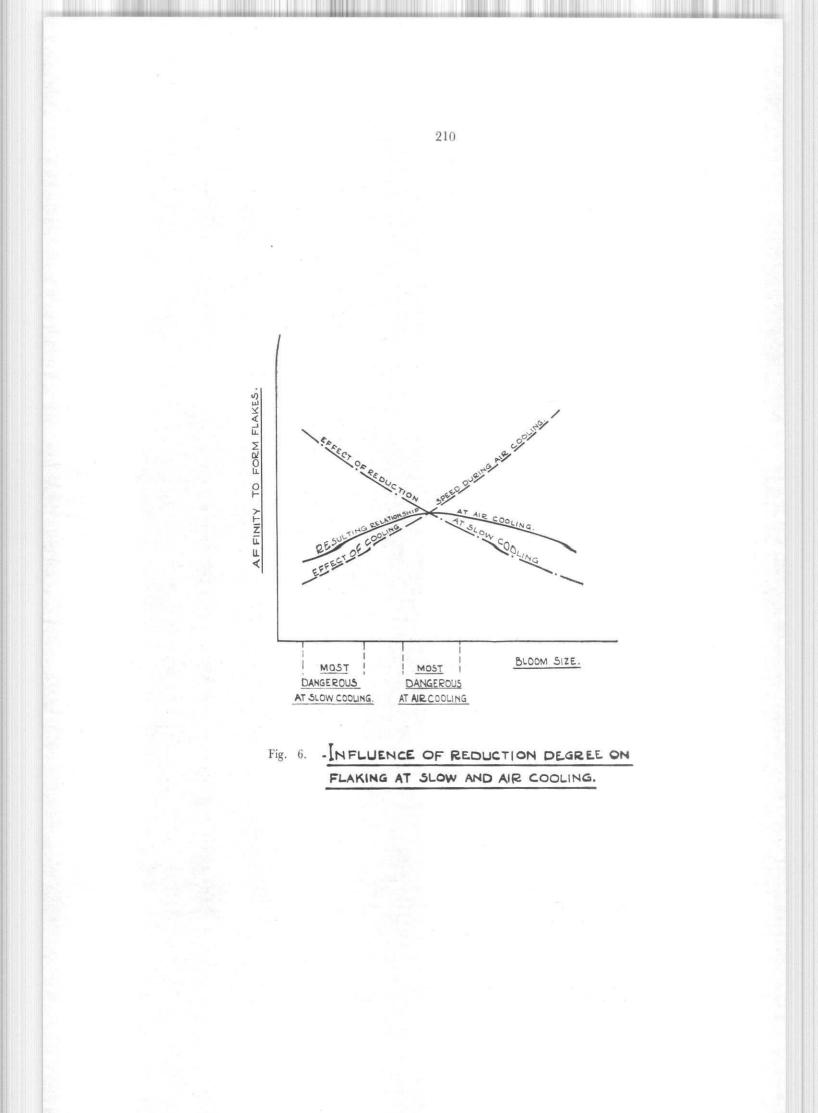
Increasing degrees of reduction during forging or rolling usually decrease the sensitivity of steels to flaking. The most dangerous degrees of reduction are from 2 to 5, i.e. when the cross section of the ingot is reduced during forging to one-half or one-fifth of the ingot size respectively. Although flakes frequently disappear with high degrees of reduction, they may ocasionally occur even after a degree of reduction as high as 100. If air cooling is done after rolling, the deteriorating effect of low degrees of reduction may be compensated by the established beneficial effect of slow cooling. Thus a bloom of 7" thickness may be less susceptible to flaking than a bloom of 3" to 4" thickness rolled from the same ingot in spite of the lower degree of reduction of the former since the latter bloom definitely cools faster. This has been expressed schematically in Fig. 6 and has to be taken into consideration when, in the current production of alloy steels, preliminary tests are made to decide whether air cooling is permissible with steels of low and medium sensitivity.

Forgings, which once have developed flakes may be made serviceable ¹,¹⁰) by reforging or rerolling. If this is done at sufficiently high temperatures and with intensive reduction, the flake surfaces get welded and the material thus is no longer sensitive to flaking during subsequent treatment. The presence of numerous flakes before this treatment may perhaps become visible in the reforged condition by a tendency of the steel to exhibit fibres in the fracture. Similarly a bloom of a sensitive steel which once has cooled slowly down to room temperature without cracking into flakes is considerably improved in sensitivity for further treatment and it may often remain free of flakes even when air cooled after hot working. This indicates that cracking into flakes and the hydrogen diffusion obtained during slow cooling result in a distribution of hydrogen and an equalization of the spots of high concentration. This is utilised in the practical procedure of forging large ingots which are repeatedly straight forged, upset forged and annealed with subsequent slow cooling. Flakes occurring during the first treatment get welded up in the second tréatment and the flaking sensitivity of the steel decreases with each treatment of this type.

There are several methods of treatment, which are useful in decreasing the affinity of steel to flaking and in avoiding the formation of flakes. These are :--

- 1. diffusion annealing at 1100 to 1200°C with soaking times of 5 to 30 hrs. and subsequent slow cooling in the furnace down to about 300°C,
- 2. slow cooling after hot working to below 200°C, the period of slow cooling being at least 20 hrs. for small blooms and of upto 100 hrs. for larger blooms,
- 3. air cooling to about 500°C, holding 5 hrs. at this temperature in a furance, heating above the transformation temperature for recrystallisation (820°C) and subsequent air cooling,
- 4. collecting the rolled or forged pieces red hot in a furnace at 500 to 600°C, heating to above the transformation temperature, air cooling to 600 to 650°C, holding 2 to 5 hrs. and subsequent air cooling.

Diffusion annealing of the ingots (heat treatment 1) may be combined with preheating for forging or rolling, but it is more effective when the ingots are allowed to cool down after annealing. The treatment most commonly used to avoid flakes is slow cooling (heat treatment 2). In forging shops, it is generally carried out in furnaces by collecting the forging at temperatures of 600 to 650°C and cooling them in the furnace to about 100°C. In rolling mills, the blooms are collected in pits and covered with ash, sand or Kieselgur. It frequently takes a week till the blooms are cooled sufficiently for removal. Comparatively extensive cooling yards are therefore required in a plant which rolls mostly alloyed and sensitive steels. If there is a shortage of space it may be sometimes helpful to pile the blooms into heaps and cover them in order to prolong the cooling period. This is permissible, however, only for materials of medium and low sensitivity, otherwise the outer blooms in the pile are in the danger of cracking. The heat treatments 3 and 4 are aimed



at shortening the furnace time and the long cooling periods. It is claimed for instance, that heat treatment 3^{33}) assures freedom from flakes in 40% of the total period and only 15 to 30% of the furnace time than that required for slow cooling. The purpose of such treatments can be understood only from the view points of diffusion and distribution of excessive local hydrogen concentrations, since these processes are accelerated in the ferritic state under increased pressures as for instance during isothermal transformation in the pearlitic range (heat treatment 4).

The rate of diffusion of hydrogen depends on differences in hydrogen concentration and on the permeability which in turn depends on temperature and pressure developed and on the structure, i.e. whether it is α or γ . The first point is obviously rather uncertain, since only the average can be judged by analysis. That deviations from the average do exist is clearly proved by the observed nonuniformities in hydrogen analyses which cannot be explained solely by losses during determination since they may considerably exceed the hydrogen content of the liquid steel. The actual figures to which the hydrogen concentrations may rise at some small spots where gas was entrapped in cavities and forced to go back into solution, when the cavities were welded up during rolling or forging, cannot be estimated properly.

The permeability is known to decrease with the temperature and is less in the γ phase as compared to that in the α phase. The representation of the rate of permeation on the logarithmical scale as dependent on the absolute temperature in order to get a straight line relationship might cause the misleading impression that the comparatively higher permeability in α iron at low temperatures is of great importance. On extrapolation to higher temperatures, the two lines in Fig. 7 ³) cross at about 1100°C where the permeabilities in the α and γ phases become equal. From the plot of the permeability values on a linear scale against temperature (Fig. 7 below) it becomes obvious that the improvement in permeation by transformation into the α phase is the highest between 1000 and 700°C and becomes negligible at temperatures below 500°C since the overall rate of permeation becomes comparatively low. The permeability of hydrogen decreases progressively with increasing carbon contents³) as can be seen from Fig. 8 showing a comparison of permeabilities during cooling of two nonalloyed steels containing 0.13% and 0.99% C. Due to the higher transformation temperature the lowcarbon steel has a higher permeability at 700 to 800°C the value of which is a little lower than that of the optimum permeation rate at 1000°C. In accordance with this, the temperature of the maximum evolution of hydrogen from steel samples soaked in hydrogen at 1200°C was reported¹¹) to coincide with the transformation temperature. In the γ range, alloying elemens do not alter the rate of permeation appreciably.¹³) For the α range, nickel and molybdenum are stated³⁴) to be without influence for carbon free steels, while chromium decreases the permeability. The smaller rate of permeation in the *c* range for medium and high carbon alloyed steels is mostly due to retained austenite.

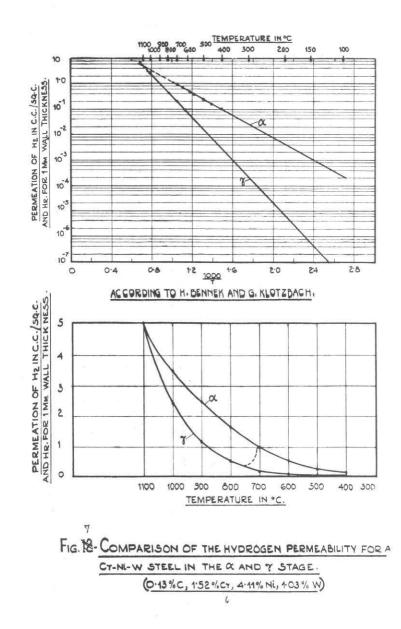
The above considerations do not take into account the additional effect of increasing pressures with dropping temperatures on the rate of permeation of hydrogen. That the permeation rate increases with pressure has been found³⁴) for a low pressure range upto 1 at. where at temperatures near 400°C, however, an incomplete absorption at the metal surface may cause an interference. If only the high temperature range from 400 to 1200°C, as used in diffusion annealing, is considered and a concentration difference of 7 to 0.5 cc/100 g is taken as the base, the acceleration of the rate of permeation due to increased pressures can be estimated from the following formula³⁴)

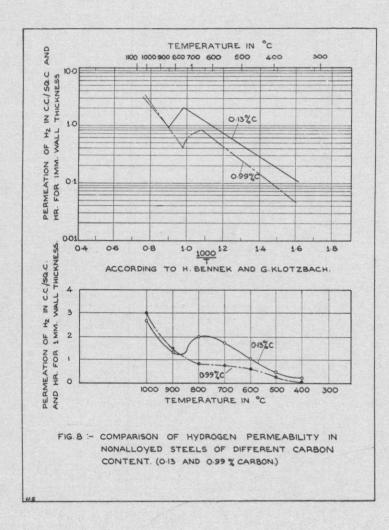
$$P = P_0 \quad (p_7 \ ^1/^2 - p_{0.5} \ ^1/^2)$$

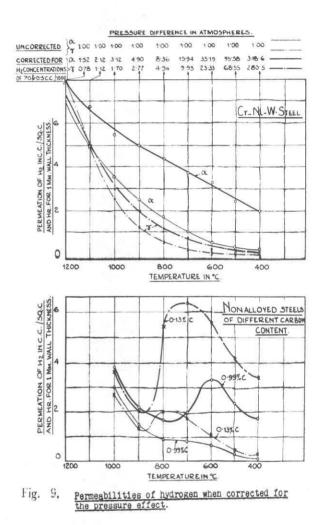
where P_0 is the rate of permeation at 1 at and p_7 and $p_{0.5}$ are the pressures developed by the corresponding hydrogen contents. The values of permeation corrected by the above calculation to include the effect of increasing pressures are shown in Fig. 9. It is clear from this figure that the permeation in γ iron is improved only slightly by increased pressures in the range from 1000 to 600°C and is even lower at higher temperatures since the pressures here become lower than 1 at. The permeation rate in α iron, however, is considerably enhanced by the effect of increased pressures are shown in the statement of the permeation rate in α iron, however, is considerably enhanced by the effect of increased pressures are shown in the statement of the permeation rate in α iron, however, is considerably enhanced by the effect of increased pressures are shown in the statement of the permeation rate in α iron, however, is considerably enhanced by the effect of increased pressures are shown in the temperatures are shown in the permeation in the permeation rate in α iron, however, is considerably enhanced by the effect of increased pressures are shown in the permeation in the permeation rate in α iron, however, is considerably enhanced by the effect of increased pressures are shown in the permeation is permeased pressures are shown in the permeased

³³⁾ O.H. Bamberger, Iron Coal Trade Rev. 148(1944) pp. 401/03.

³⁴⁾ P.L. Chang and W.D.C. Bennett, J. Iron & Steel Inst. 170 III(1952) p. 205.







sures, particularly in the range from 800 to 600°C where the transformation takes place usually. Furthermore the rate remains remarkably high even at low temperatures upto 400°C.

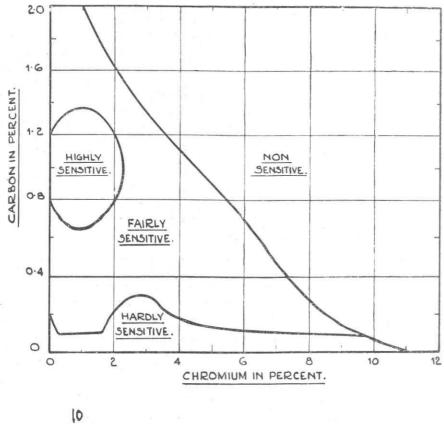
Having separated the effects of temperature and pressure on the rate of permeation, it now becomes understandable that diffusion annealing in the range of 800 to 600° C, if it is done after transformation into the α stage, may be as good or even more effective than diffusion annealing at 1100°C in the γ stage. This is mainly due to the effect of pressure. Diffusion in the α range has the advantage, that it may be prolonged easier without the appreciable deterioration which may occur during high temperature heating and it may be continued during the period of slow cooling. It is further evident from Fig. 9 that high transformation temperatures as obtained for instance in low carbon or in high chromium steels provide the opportunity for making use of the accelerated rates of permeation in the α stage due to the higher pressures.

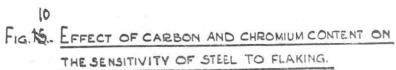
6. Steel Composition and structure as affecting the flaking sensitivity

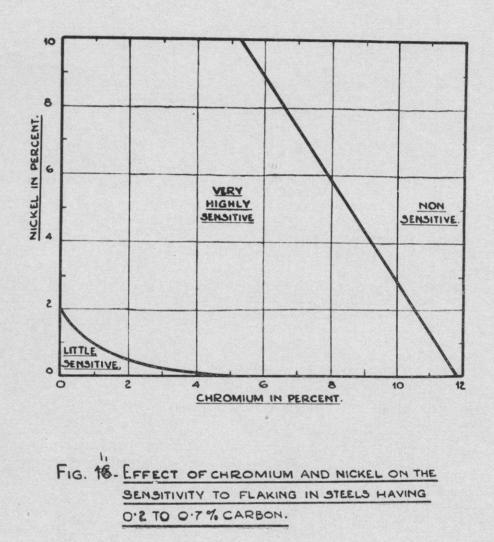
It is the general rule that all alloying elements, which decrease the temperature of martensite formation, and increasing carbon contents favour the occurrence of flakes. But there are some limitations to this generalization. It is rather difficult to indicate the limits accurately, since they change in relation to the other factors of influence and it is also difficult to give a complete account because of the large variety of steels to be considered. It may be stated, however, that the susceptibility to cracking is very small in nonalloyed steels containing less than 0.2 % C and that the sensitivity increases with the carbon content upto a maximum at 0.8 at 1.2 % C. Such steels may be fairly sensitive, particularly if they are produced to high purity in basic electric furnaces. The following classification may be made as regards the effect of additional alloying elements :—

- 1. High sensitivity may be observed in nickel steels with more than 3 % Ni and chromiumnickel steels with more than 2 % Ni and 1.5 % Cr. For the most susceptible steels with 4.5 % Ni plus 1.5 % Cr or 2 % Ni plus 2.5 % Cr, particularly when containing additions of molybdenum or tungsten, lower carbon contents of even 0.2 % C decrease the sensitivity only slightly.
- 2. Medium sensitivity is encountered in nonalloyed steels having 0.8 to 1.2 % C, steels of the same carbon content having 1 to 2 % Cr and medium carbon constructional steels containing 1 to 2 % Cr or Mn. The sensitivity is further increased by additions of molybdenum or silicon.
- 3. Low but a noticeable sensitivity is found in non-alloyed steels having 0.5 to 0.7 $\frac{0}{0}$ C, case hardening steels with alloy contents of about 1 $\frac{0}{0}$ Cr or Ni and spring steels with the exceptions of the chromium-vanadium and silicon-manganese types. The last two types may be added to the previous second group.
- 4. Hardly any sensitivity is observed to occur in mild steels with less than 0.2 % C, low alloy structural steels, aluminium containing steels like the nitriding steels with about 1 % Al and in chromium-vandium steels with about 0.3 % C and 1.5 to 2.5% Cr.
- 5. Nonsensitivity is found to occur in practice for air hardening and high alloyed steels like steels containing 12 % and more chromium, high speed steels, high-tungsten hot die steels, and all the austenitic chromium-nickel and manganese steels.

Generally speaking a ledeburitic structure diminishes the susceptibility to flaking. The same is true for a ferritic structure in steels which do not undergo transformation. Furthermore, flakes occur very rarely in steels having the austenitic structure. Under exceptional conditions like for instance by soaking in a hydrogen atmosphere and quenching it is possible⁴ to produce flakes in ferritic 4% Si transformer steels. Occasionally flakes may be formed in austenitic 18/8 chromium-nickel or 12% managanese steels as the result of atomic arc welding in a hydrogen atmosphere. An attempt is made to represent the above findings graphically. Fig. 10 shows the dependence on carbon and chromium contents and Fig. 11 shows the extensive







field of high sensitivity in the chromium nickel steels. The diagrams are based on general experience and the lines cannot be claimed to be accurate since, as explained, so many additional influencing factors play their part thereby altering the picture. Therefore only a rough idea may be presented in these figures.

In light of the foregoing discussions regarding gas evolution during solidification and the permeation of hydrogen during heat treatment, it is now possible to offer some explanations about the inherent reasons of the different behaviour of several types of steel as regards their sensitivity to flaking. Steels which solidify into the primary structure of δ iron such as low-carbon steels 4% silicon steel, high chromium stainless steel and even austenite chromium-nickel steels of the 18/8 type, ³⁵ are found to lose more hydrogen during solidification since the hydrogen evolution occurs at a lower hydrogen content. Even if steels like those containing high chromium and high nickel pick up more hydrogen during melting, as may be concluded from their generally higher hydrogen content in the liquid state 13 and from the increase of solubility by nickel in the liquid¹⁶ and solid 36 state, they are more capable of expelling this hydrogen. Thus they retain in the solid state a lower concentration than that retained by steels solidifying into the γ structure. This may be derived from Fig. 3 if it is sassumed that the cohersive strength of the solidifying steel is about 0.013 tons/sq. in¹⁹. at 1535°C. Then the evolution should be expected to occur at 12.2 cc in the \$ range and at 17.7 cc/100 g. in the γ range. In the case of steels which solidify in the γ range, the solidification temperature is important. If this temperature is low, for instance only 1350°C, hydrogen evolution may take place even at 14 cc/100g which means that less hydrogen remains entrapped during solidification. All high carbon steels such as a nonal-loyed steels with 2.3% C, high chromium tool steels with 1.5 to 2% C and 12% Cr, high speed steels, hot die steels with 9% W and even the 12% managanese steels belong to this group where solidification occurs at lower temperatures and therefore the amount of hydrogen entrapped is less.

In explaining the effect of pressure on the rate of hydrogen permeation it was clarified, that the nonuniformities in hydrogen concentration in steels with high transformation temperatures can be more easily evened out by permeation particularly if they are annealed in this high temperature range and subsequently slow cooled. This is true, for instance for the high speed and high chromium steels. Furthermore, steels like the chromium stainless steels and the aluminium containing nitriding steels and also to a certain extent the austenitic 18/8 chromium nickel steels go through the δ range during heating the ingots to high temperatures and thus undergo during the usual treatment conditions favourable for an equalisation of hydrogen contents.

If steels like the two austenitic steels (18/8 Cr Ni and 12% Mn), a ferritic steel (4% Si), a stainless chromium steel (15% Cr) and some medium carbon alloy or nonalloyed steels are soaked in hydrogen at 1100°C in sections of 2.1/2" sq. and water quenched, then the thermal stresses due to quenching should be the same in all the cases. Moreover, in the forged condition of the samples, the influences of enrichment by segregation and equalisation by permeation are eliminated. The 4% silicon steel is soaked in the α stage, and the same phase is present partially in the 15%chromium steel. The solubility of hydrogen is lower in α iron (vide Fig. 2). Further it is known that silicon diminishes¹⁵) the solubility of hydrogen in iron and that chromium decreases³¹) the rate of permeation in α iron. Therefore the absorption of hydrogen in these two steels should be lower. The silicon steel is stated⁴ to crack into internal flakes after prolonged soaking of 60 hours, while the medium carbon steels crack even within 10 hours. The austentite steel and the 15% chromium steels do not crack even after prolonged soaking. The medium carbon and the 15% chromium steels build up additional stresses due to transformation and hardening. The latter steel, however, does not crack*since it contains less hydrogen. Of the steels which do not have a transformation, only the silicon steel is found to crack, whilst the austenitic steels, which should contain more hydrogen, do not crack. The silicon steel becomes very coarse and brittle during treatment, and this makes it more sentsitive to cracks than the chromium steel in spite of the hardening effect in the latter. This appears to confirm the opinion that brittleness

³⁵⁾ H. Wentrup & H. Schrader, Arch Eisenhuettenwes, 20 (1949) p. 173.

³⁶⁾ C.A. Zapffe and C.E. Sims, Trans. A.I.M.E. 145(1941) p. 225.

constitutes an additional factor favouring crack formation and ductility, which is a significant property of the austenitic steel, is a factor preventing flake formation.

7. THE INFLUENCE OF SEVERAL REMAINING FACTORS AND THEIR CO-OPERATIVE EFFECT.

A dirty steel containing excessive amounts of slag inclusions may be found occasionally to be defective because of the additional presence of flakes. The flakes formed in this case frequently are smaller in size and are more distributed than in a pure steel. The inclusions, which represent spots of low strength as compared to the overall cohesion strength of the material, may act as starting points of the ruptures, if present together with a high hydrogen content. The fact, however, that steels of high purity such as the basic electric furnace steels are found to be particularly inclined to flakiness, proves that there is no actual relation between the degree of impurity of steel and its affinity to flaking.

Since the segregation of hydrogen during the solidification of an ingot may cause an enrichment of the mother liquor in hydrogen, it is natural that the segregated portions are more likely to form flakes. Therefore, greater accumulations of such internal fissures may be observed in the vicinity of segregations and particularly of pipe segregations. If, however, the steel suffering from segregation is treated to equalize the hydrogen distribution, for instance, by diffusion annealing and forging, the segregated portions are no longer more sensitive to flaking than the surrounding material. This has been demonstrated by soaking in hydrogen and subsequent quenching.⁵ It means that higher flaking susceptibility of the segregated portion is not due to accumulation of impurities as such but that it is caused by hydrogen enrichment which is bound to occur simultaneously with the local enrichment of other segregating elements.

The temperature of flake formation during cooling of forgings was determined¹ to lie between 100 to 300°C for a ball bearing steel contianing 1.4 % Cr, and between 100 to 200°C for a chromium-nickel steel containing 3.5 % Ni. Therefore, the temperature of flake formation was concluded to be near 200°C. From the calculations of pressures developed by different hydrogen contents in excess of that due to 0.5 cc/100 g., at which concentration the pressure was found to be insufficient for disruption, it is possible to estimate the temperatures at which the tensile strength of a steel is just exceeded. If the tensile strength of a mild steel is taken to be 32 tons/sq. in and that of an air cooled chromium-nickel alloy steel as about 85 tons/sq. in., the hydrogen contents at which the excess pressures cross the horizontal lines representing the two tensile strengths may be plotted against the temperature of intersection. Now it becomes evident that the high tensile steel should be expected to crack between 100 and 270°C and the low tensile steel should crack between 120 and 300 °C and that the temperature of cracking is lower for smaller hydrogen concentrations. This result of the theoretical calculations is in excellent agreement with the experimental determinations of the temperature of cracking.

From the two lines for selected tensile strengths the contribution of some other influences may be recognised simultaneously. The presence of soft spots, for instance of ferrite in a banded structure, should create conditions which correspond to those for the lower tensile strength and thus it is clear that such a structure increases the susceptibility to flaking, since a lower hydrogen content is required for cracking. Further, the additional effect of residual stress can be seen from the dotted line in the diagram drawn to show that the presence of this stress shifts the strength of the steel towards that of the low tensile steel. The value of 10 tons/sq. in., as considered here, is rather high in comparison to that to be expected normally after slow cooling. Quenching and particularly transformation stresses, however, as caused by quick cooling may rise to much higher values. The representation gives a fairly clear idea that due to the co-operation of several factors like bands of soft ferrite and residual stresses, the hydrogen contents required for flaking may become rather low and that about 2cc/100g may be theoretically enough to produce flakes under unfavurable conditions.

It has been reported repeatedly that quenching of steel enriched in hydrogen may suppress flake formation and that the flakes are produced later after a certain period of incubation, either at room temperature¹⁰ or during heating.²,¹⁰ An explanation³⁷ useful in understanding this time lag is that a certain time is necessary before the atomically dissolved hydrogen can come out of solution, form molecules, and develop the pressures required for rupture. The incubation period is noticeable, however, only in exaggerated cases. The period may be considered to be almost over if rolled or forged billets or blooms are cooled either in air or even slower.

The opinion ¹³ that no steelmaker or consumer has ever been seriously troubled by cracks produced by the absorption of hydrogen in a solid state and quenching does not take into account that such cases have been actully reported³⁸ to cause defects in case hardened rings. Here, carburising of deep cases in salt baths was found to cause flakes which were formed during hardening. To avoid such cracking, provisions had to be made to keep the carburising salt baths free from water. Hardening stresses, increased by a deep case as compared to a small wall thickness, played an additional part since quenching in salt baths, maintained at 220 °to 230 °C, did not produce flakes. This was confirmed³⁹ for carburisation in solid media (charcoal: barium carbonate as 60: 40) and it was shown that the original hydrogen content may be of importance since a low hydrogen steel containing only 1cc 100g. did not get flakes when carburised at 1010°C, air cooled and quenched from 840°C in oil, while a steel of the same composition but containing 3.78 cc/100g cracked into typical flakes during the hardening quench. Such cases which are fortunately rare, prove that a high hydrogen steel, which by proper control during production remained free of flakes, may develop flakes if the hydrogen content is increased by absorption during a later heat treatment and if other additional conditions such as quenching and hardening stresses favour the formation of flakes.

8. SUMMARY

1. A fairly complete review has been worked out about the factors influencing the formation of flakes in steel, summarising the main principles of upto date practical experience and important theoretical considerations.

2. The main reason for flaking, as generally accepted, is the pressure developed by hydrogen coming out of solution. In addition, other factors such as stresses, local concentrations and brittleness also play a part. The pressures developed by hydrogen contents varying from 0.5to 12 cc/100g have been recalculated for a temperature range from 100 to 1650°C.

3. Regarding the effect of steel making, the established knowledge that steels made by acid processes are less susceptible to flakes is in agreement with the lower hydrogen contents of liquid acid steels. The furnace atmosphere is of little influence. The lime additions and the moisture content of the lime, however, are important in increasing the hydrogen content of basic steel. Basic slags have an higher hydrogen content as combined into water than acid slags. The hydrogen content of liquid steel decreases during boiling and oxidising conditions. A highly deoxidised and inactive steel bath is very sensitive to the pick up of hydrogen.

4. Hydrogen absorption may occur during tapping if runners and ladles contain remnants of moisture. Killing by aluminium in the ladle usually diminishes hydrogen by evolution. Top pouring makes the steel more susceptible to flaking than bottom pouring. Coating increases the sensitivity to form flakes if it is done with hydrogen containing agents such as tar and lacquer in thick layers and on cold moulds. The shape of the ingot section is stated to have no influence. The affinity to flaking increases, however, with the height and the weight of the ingots. Hydrogen segregations cause enrichments in the core and the top of the ingots.

5. Increasing degrees of reduction during hot working reduce the sensitivity of steels to flaking. Welding of flakes by reforging results in non-susceptibility. The most common treatment to avoid flakes is slow cooling. Some other heat treatments meant for shortening the cooling periods aid at an equalisation of the differences in hydrogen concentrations by diffusion. The

- 37) Suggested by Honda and Hirone, Science Reports Tohoku Imp. University 25(1936) Dec. pp. 713/725.
- 38) H. Diergarten, Stahl and Eisen 608(1940) pp. 1027/1035.
- 39) H. Schrader, discussion to 34, vide also 5.

rate of permeation of hydrogen is dependant on the temperature, the pressure developed and on the structure *i.e.*, whether it is α or β . The influence of each factor was separated and it was shown, that the higher rate of permeation in the α structure and the high pressures are prerequisites to get the most effective conditions for equalisation.

6. A general review of the sensitivity of steels having different compositions and structures in respect to their tendencies to flaking was presented. An explanation about the experienced differences was offered on the basis of the ability to expel hydrogen during solidification and to diffuse hydrogen during preheating and cooling. For this purpose, a solidification into the δ structure and high transformation temperatures and the existence of the α structure at high temperatures are favourable. As an additional factor, ductility may come in particularly in the austenitic steels.

7. The extent to which impurities and segregations may contribute was considered. The temperatures of flake formation between 100 to 300°C were shown to be derivable from the calculated pressures exceeding the cohesion strength. The relative and cooperative effects of hydrogen contents, tensile strengths and residual stresses were compared on the basis of the calculated results. Finally, some deviations from common experiences such as the suppression of flakes by quenching, which develop later during aging or heating, and the formation of flakes due to hydrogen absorption during carburising and occurring during the hardening quench were reported.

Hydrogen content cc/100g in Open Hearth Elec. Arc			Author	Method of
			Sampling	
Acid	Basic	Basic		
6.5 (10)	9.4 (19)	9.0 (8)	H. Wentrup, H. Fucke, O. Reif.	Suction method
4.2 (11)		6.2 (14)	C.Sykes, H.H. Burton, C.C. Gegg.	Pencil method.
3.5 (3)	7.0 (5)	5.1 (7)	R.M. Cook, J.D. Hobson.	Pencil method.
3.8 (3)	7.2 (5)	4.6 (7)	do.	Sealed mould method.
3.4 (3)	5.1 (1)	3.1/5.5(2)	D.J. Carney, J. Chipman, N.J. Grant	Suction method.
3 to 6	5 to 12	4 to 10	J. Bletin, S. Mischosniky, G. Coin, P. Bastin.	Pencil method.

Table 1—Average content of hydrogen determined for different steel making processes before tapping with different methods of sampling.

Number of casts in breakages