

LACK OF HOMOGENEITY IN LARGE INGOTS.

In No. 3 "Papers on Engineering Subjects" of April, 1922, there is a paper entitled "Some Remarks on the Manufacture of Steel Forgings" in which the Author mentions the lack of homogeneity of ingots in general, implying however, that with sufficient care and precautions this difficulty can be eliminated. It is the object of this paper to point out that this is not the case at the present time and to give some idea of the limits of accuracy which can reasonably be expected when dealing with ingots of large size.

The great difficulty involved in producing ingots of large size is at the root of nearly all the trouble inherent in the production of the many large steel forgings, which are so essential a factor in the design of modern large power units, and it is a fact that in many cases specifications for steel for large forgings are made out with a total disregard of the limits of accuracy beyond which it is impossible to work in the production of the initial ingot from which the forging is to be produced.

This question is of so great an importance both to the steel maker and to the engineer that the "Iron and Steel Institute" were led in 1924 to set up a committee to study the problems of inclusions in steel and of the heterogeneity of steel ingots. The Report of this committee was presented at the Annual Meeting of the Iron and Steel Institute in May, 1926, and takes up some hundred pages, from which it is proposed to extract some facts and figures which it is thought will be of interest and service to all Engineer Officers.

The committee dealt with ingots of steel only, varying in weight from 15 cwts. to over 172 tons, the number of Ingots actually examined being 16. It may be thought that this is a small number from which to draw deductions of practical value, but in view of the extreme care with which the examinations and investigations were made, this objection is hardly tenable even if it cannot be dismissed entirely.

As a first step each ingot was sectioned along its axis, the machined surface being then ground and polished as required. The examination of a surface prepared in this manner does not, as a rule, reveal anything further than the existence of the pipe cavity at the top of the ingot, accompanied possibly by slight minor cavities and looseness along the central axis.

After this preliminary examination the prepared surface was "etched" with a suitable etching reagent in order to obtain evidence as to, what may be termed, visible heterogeneity. In addition to this direct inspection, the Baumann sulphur printing method was employed in order to show up any inequality in the distribution of the sulphur compounds over the polished surface.

The value of examination of steel surfaces by means of sulphur prints is well understood and increasing use is being made of this method, as no substitute has yet been found which is

equally harmless, reliable and valuable; it has even been stated in support of this method of examination, that if a sulphur-free steel is ever prepared, then a small proportion of that element should be added in order to permit of sulphur prints being taken.

It is not possible in a paper of this nature to give the full details of all the examinations that were made, but it is proposed to give a few typical results, in order to draw attention to the order of the errors which may be expected in certain given cases. In order to do this a table has been prepared giving the following data from typical ingots, viz. :—size and weight, “Cast analysis,” and the highest and lowest content of each of the principal elements contained in the ingot, as obtained from the prepared surfaces.

When examining the figures in the accompanying Table I, it must be remembered that the upper portion of the Ingot has in all cases been discarded, and that the results shown are those taken from what may be termed the working portion of the Ingot.

As might be expected, the results in general show that the larger the Ingot, the greater the divergence of the maximum and minimum figures from those of the test figures. The greatest differences are to be found in the figures obtained for the Carbon, Sulphur and Phosphorous determinations, and these are unfortunately the three most important elements from the point of view of the normal specification.

The causes which lead to heterogeneity are many, it being extremely difficult to distinguish between the various influences at work, or to determine their comparative effects, but the following observations may be of interest as indicating the trend of the ideas put forward by the members of the committee on this subject :—

(a) Steel is not a simple metal; in its liquid form it is a solution of other elements or of their compounds in iron. When solidification begins the primarily formed crystals consist of solid solutions of these same elements (or of their compounds) in Iron, but, owing to selective freezing they are of a greater purity than the original liquid. The remaining liquid steel must therefore become progressively richer in these compounds, and is probably of lower density than the original steel.

(b) In the past it has been widely held that heterogeneity was associated with rapid cooling, and that the slower the speed of cooling, the more uniform would be the composition of the solidified ingot.

It may, however, be shown, by consideration of the equilibrium diagrams of the various elements concerned, that in fact the opposite is the case: rapid cooling provides insufficient time for the complete separation of the phases, and thus results in a more homogeneous product.

TABLE I.

RANGE OF COMPOSITION, AS OBTAINED BY ANALYSIS OF DRILLED SAMPLES.

The figure of "range per cent." for each ingot and element is expressed as a percentage of the element as ascertained by analysis of the corresponding test ingot. The ingots are arranged in order of increasing weight.

Weight of Ingot.	Carbon.				Manganese.				Silicon.				Sulphur.				Phosphorous.			
	Test Ingot.	Maximum.	Minimum.	Range per cent.	Test Ingot.	Maximum.	Minimum.	Range per cent.	Test Ingot.	Maximum.	Minimum.	Range per cent.	Test Ingot.	Maximum.	Minimum.	Range per cent.	Test Ingot.	Maximum.	Minimum.	Range per cent.
Tons. Cwts.																				
1 5	0.52	0.54	0.48	12	0.84	0.84	0.79	6	0.21	0.22	0.20	10	0.037	0.040	0.032	22	0.036	0.038	0.032	17
2 10	0.41	0.46	0.40	15	1.06	1.04	0.98	6	0.52	0.52	0.50	4	0.056	0.057	0.044	23	0.052	0.062	0.047	29
8 0	0.39	0.45	0.31	35	0.96	1.04	0.95	9	—	—	—	—	0.024	0.029	0.016	54	0.025	0.031	0.023	32
10 10	0.30	0.34	0.21	43	0.74	0.77	0.68	12	0.14	0.14	0.13	7	0.017	0.026	0.009	100	0.010	0.011	0.009	29
24 17½	0.44	0.49	0.35	32	0.78	0.80	0.73	9	0.29	0.29	0.27	7	0.033	0.048	0.025	70	0.042	0.042	0.030	29
64 0	0.40	0.50	0.33	43	0.63	0.69	0.63	10	0.16	0.17	0.16	6	0.035	0.050	0.029	60	0.038	0.050	0.026	63
110 0	—	0.37	0.15	—	—	0.71	0.63	—	—	0.23	0.20	—	—	0.059	0.021	—	—	0.050	0.024	—
150 0	0.33	0.55	0.19	109	0.79	0.92	0.73	11	0.13	0.14	0.11	23	0.030	0.080	0.018	206	0.033	0.090	0.023	203

The importance of the above conclusions is evident if we imagine the sequence of the events which take place as an ingot solidifies. In all ingots the outer layers pass through the freezing range very rapidly on account of the great absorption of heat by the mould, there being therefore insufficient time for the phases to separate; hence the crystals of steel formed in the outer layers show little or no variation in composition, and there is little segregation.

After the initial "chilling" effect of the mould has been overcome, the rate of cooling will gradually decrease, the time occupied in passing through the freezing range thus increasing and the variations in composition becoming more marked.

In order that the material in the centre of the ingot may cool it must dissipate its heat by conduction through the surrounding steel to the outside of the mould, whence the heat is finally rejected, mainly by radiation. As heat cannot pass from one body to another unless the latter is at a lower temperature than the former, it is evident that a gradient of temperature must exist across any section of the ingot. It is probable, as a little thought will show, that this temperature gradient is steeper at the edges than in the centre of the ingot, and that the rate of cooling is therefore somewhat more rapid as the exterior of the ingot is approached.

It is suggested, therefore, that in the central zone the separation of the phases will be most complete and the crystals will show the lowest percentages of the segregating elements. Further, it will be evident that the cross section of the ingot must exercise a fundamental bearing upon the process: the greater the diameter the slower will be the rate of cooling of the interior portion of the ingot, and therefore the more marked will the differences in composition become.

From these considerations it appears that even when steel is made under the most perfect conditions, absolutely free from any extraneous material and of uniform composition when entering the mould, it must exhibit, when solid, certain variations of composition because of this selective freezing. The degree to which these variations manifest themselves will increase:—

- (1) As the carbon and phosphorous content of the steel increases;
- (2) As the rate of freezing of the ingot falls; and
- (3) As the cross section of the ingot increases in size.

The factors governing the segregation of sulphur require separate consideration, but, so far as can be ascertained from the studies already made on the question, it is very difficult to assign any definite composition to the sulphide which is present in liquid or solid steel.

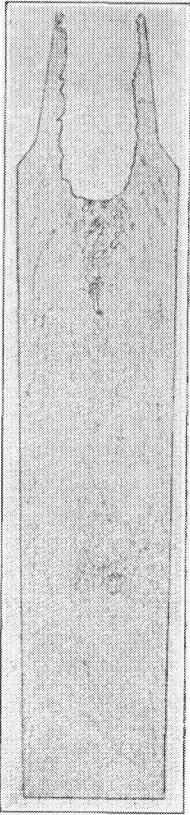


FIG. 1.—15-Cwt.
Ingot Sulphur Print.

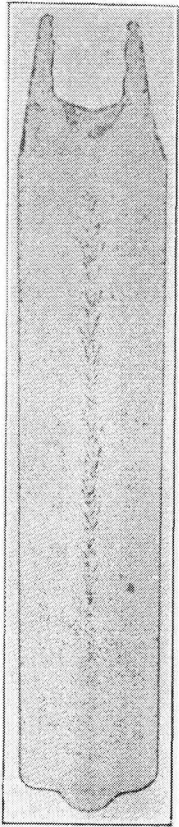


FIG. 2g.—1¼-Ton
Ingot Sulphur Print.

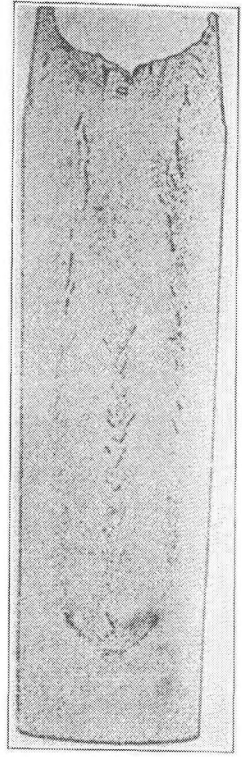


FIG. 3g.—2½-Ton
Ingot Sulphur Print.

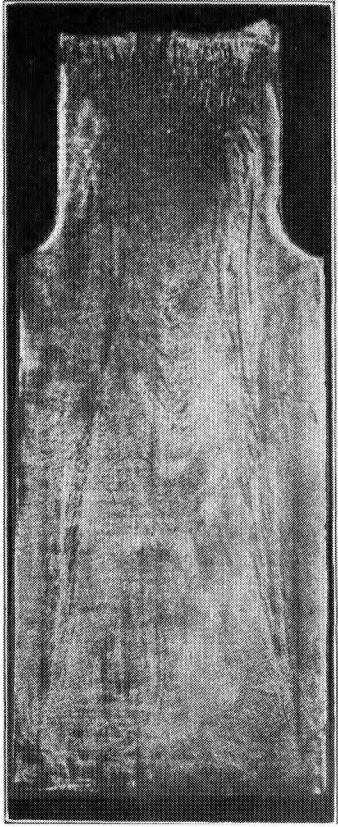


FIG. 10f.—10½-Ton
Ingot Sulphur Print.

When manganese is added to the steel in the bath, the distribution of the sulphur between the two elements iron and manganese is determined by the relative amounts of the two metals present and their respective affinities, the resulting sulphide being always of a complex character $(\text{FeMn})\text{S}$. It is generally accepted that this complex sulphide is insoluble in liquid iron, and its distribution is governed by a different law from that of the other constituents: in addition, there is no doubt that this sulphide is of lower density than liquid iron. It has been suggested that there are reactions between these sulphides and the oxides left in the metal, as they are sometimes found to be associated together in the segregated areas.

Sulphur, then, mainly exists in the liquid steel in the form of minute globules of an immiscible, complex sulphide. During the rapid formation of the columnar crystals in an ingot, these globules are pushed farther into the interior of the ingot by the glowing crystalites, and as a result there is an increase of the sulphur content in the layer of mother liquor at the face of crystallisation. The small globules coalesce and comparatively large masses collect at the tips of the columnar crystals. These masses by virtue of their lower density commence to rise, but are entangled by the glowing crystals, thus forming zones of segregate, which are found as string-like masses at the surfaces of the columnar crystals.

In the mass of the still liquid steel of the ingot a similar process occurs, the small globules of sulphide coalescing and tending to rise, the larger the globule the quicker being the rate of rising. The globules from the central or axial parts of the ingot rise into the upper portion and ultimately into the head, the lower portion of the ingot being left with much less sulphur than any other part, this being especially so in the central zone. In the intermediate zones, between the columnar crystals and the axial zone, the sulphur segregate will not be able to rise as far as the head before it is trapped by the advancing wall of the ingot. The particles in the outer zones will have but a small period of time in which to rise, and therefore only travel a short distance before the thickening wall intercepts their further progress. The nearer the particles are situated to the axis of the ingot, the longer periods will they have for rising, and therefore the farther will they proceed on their journey towards the head before being arrested by the solidifying steel shell of the ingot.

If this picture is correct, then it would be anticipated that the lines of segregate or "ghost lines" would show a gradual taper inwards as they pass from the bottom corners of the ingot towards the head, and that the degree of segregation would become more pronounced as the head is approached. Such is found to be the case, as is shown by the accompanying sketches.

Analysis of steel ingots and forgings.—As steel articles are in general ordered to a specification giving the desired chemical analysis, it may be of interest to consider what is the value of the results of such analysis.

From what has been said it may well be appreciated that a single sample taken from a steel forging cannot be regarded as representative of the composition of the whole, since it is a physical impossibility with the present methods to produce a perfectly homogeneous ingot. Further, the segregates in steel are often very minute, and thus even two samples from one drilling cannot be considered as being entirely homogeneous.

Regarding the chemical analysis itself, the figures given for the various determinations of carbon, sulphur, &c., cannot be taken as being of invariable accuracy. The chemical determination of the elements present in any material is subject to the ordinary laws as to the limits of accuracy to which these processes may be considered as reliable.

Unfortunately it is the habit of analytical chemists to report their results to three and sometimes four places of decimals, and this has undoubtedly given rise in many quarters to the belief that the results may therefore be considered as accurate to the last published place. This has been a frequent cause of friction in the accepting or rejecting of material supplied to a definite specification.

Check analyses of any given specimen by different chemists will often vary to a considerable degree, due not only to the personal equation but also to the almost inevitable variations in portions of the same sample.

As an instance of this, the analysis of a typical nickel steel pinion shaft which came under the author's notice is given, as reported by the firm who made the forging, and as reported by the Admiralty Chemist at Portsmouth.

<i>Element.</i>	<i>Firm.</i>	<i>Portsmouth.</i>
Carbon - - - - -	0·30	0·320
Silicon - - - - -	0·18	0·210
Sulphur - - - - -	0·029	0·030
Phosphorous - - - - -	0·031	0·018
Manganese - - - - -	0·57	0·590
Nickel - - - - -	3·62	3·695

The above figures were not selected as being in any way out of the ordinary with regard to their divergence, and as many far more striking instances could be produced in almost any chemical laboratory, or overseer's office.

In order that some idea may be obtained as to the reasonable limits of accuracy in chemical analyses the following figures are given. (Authority, Bauer and Deiss.)

TABLE II.

Carbon.—If the carbon determination be carefully carried out the following values may be taken as permissible variations in the analysis of samples weighing about 3 grms.

With a carbon content.		Greatest allowable
From	To	Deviation.
0.02	0.15%	$\pm 0.005\%$
0.15	1.00%	$\pm 0.01\%$
1.00	2.00%	$\pm 0.02\%$
2.00	at higher per cent.	$\pm 0.03\%$

These figures apply only to direct combustion methods.

Barium Hydroxide Method (below carbon steel = 0.6%).

Carbon Content.		Permissible Deviation.
0.01	to 0.25%	$\pm 0.002\%$
0.25	„ 0.60%	$\pm 0.004\%$

Silicon.

Si Content.		Allowable Error.
0.01	to 0.25%	$\pm 0.005\%$
0.25	„ 1.00%	$\pm 0.01\%$
1.00	„ 5.00%	$\pm 0.02\%$
5.00	„ 10.00%	$\pm 0.03\%$

(The above refers to Material Soluble in Acid.)

Si Content.		Allowable Error.
1	to 10%	$\pm 0.05\%$
10	„ 20%	$\pm 0.10\%$
20	„ 50%	$\pm 0.15\%$
50	„ 100%	$\pm 0.2\%$

(The above refers to the ignition method.)

Manganese.

Manganese Content.		Allowable Error.
0.0	to 0.2%	$\pm 0.01\%$
0.2	„ 1.0%	$\pm 0.02\%$
1.0	„ 5.0%	$\pm 0.04\%$
5.0	„ 10.0%	$\pm 0.06\%$
10.0	„ 30.0%	$\pm 0.08\%$
30.0	and upwards	$\pm 0.10\%$

(This applies to the gravimetric method.)

Manganese Content.		Allowable Error.
1	to 2%	$\pm 0.04\%$
2	„ 10%	$\pm 0.06\%$
10	to upward.	$\pm 0.1\%$

(It is difficult to get good results with nickel steel with this method.)

Phosphorous.

<i>Phosphorous Content.</i>	<i>Allowable Error.</i>
0. to 0.03%	$\pm 0.001\%$
0.03 " 0.1%	$\pm 0.002\%$
0.1 " 0.2%	$\pm 0.003\%$
0.2 " 1.0%	$\pm 0.005\%$
1.0 " 2.0%	$\pm 0.01\%$
2.0 " 5.0%	$\pm 0.02\%$
5.0 at more	$\pm 0.05\%$

(This applies generally to all methods of analysis.)

Arsenic.

Content up to 0.3% Result to $\pm 0.01\%$

Sulphur.

<i>Sulphur Content.</i>	<i>Allowable Error.</i>
0.2 to 0.4%	$\pm 0.005\%$
0.015% at less.	$\pm 0.003\%$

Copper.

Copper content less than 0.2%. Allowable Error 0.005%.

Nickel.

<i>Nickel Content.</i>	<i>Allowable Error.</i>
0.0 to 1.0%	$\pm 0.005\%$
1.0 " 5.0%	$\pm 0.02\%$

(This applies to Dimethylglyoxime method or electrolysis.)

Chromium.

<i>Chromium Content.</i>	<i>Allowable Error.</i>
0 to 0.5%	$\pm 0.005\%$
10% and above.	$\pm 0.1\%$

Aluminium.

<i>Aluminium Content.</i>	<i>Allowable Error.</i>
0. to 1%	$\pm 0.05\%$

Titanium.

<i>Titanium Content.</i>	<i>Allowable Error.</i>
0.05 to 0.2%	$\pm 0.02\%$
0.2 " 1.0%	$\pm 0.03\%$
1.0 " 5.0%	$\pm 0.1\%$
5.0 " 10.0%	$\pm 0.2\%$

Iron. (In special Steel, and Ferro Alloys.)

<i>Fe. Content.</i>	<i>Allowable Error.</i>
0.1 to 0.5%	$\pm 0.02\%$
0.5 " 2.0%	$\pm 0.03\%$
2.0 " 10.0%	$\pm 0.05\%$
10.0 " 25.0%	$\pm 0.10\%$
25.0 " 50.0%	$\pm 0.15\%$
50.0 " 100.0%	$\pm 0.20\%$

Tungsten.

<i>W. Content.</i>	<i>Allowable Error.</i>
0.2 to 0.5%	± 0.02%
0.5 „ 1.0%	± 0.03%
1.0 „ 5.0%	± 0.05%
5.0 „ 10.0%	± 0.08%
10.0 „ 20.0%	± 0.10%

The following table of Tolerances in chemical analysis which was put forward by C. H. Risdale, F.I.C., F.C.S., before a joint meeting at Middlesborough, of the Cleveland Institution of Engineers, the Newcastle section of the Society of Chemical Industry, and the Institute of Chemistry, is also added as bearing on this point.

TABLE III.

Composition.

	<i>Actually found under exceptionally favourable conditions.</i>	<i>Suggested.</i>
<i>Carbon, up to</i>		
0.35%	0.025%*	0.015%
0.36-0.75%	0.033%	0.020%
0.76-1.20%	0.05%	0.025%
<i>Silicon, up to</i>		
0.10%	0.025%	0.015%
0.10-0.20%	0.032%	0.020%
<i>Sulphur, up to</i>		
0.08%	0.0077 (0.008)%	0.005%
<i>Phosphorous, up to</i>		
0.05%	0.0062%	0.005%
over 0.10%	0.012%	0.007%
<i>Manganese :—</i>		
<i>Plain, carbon</i>		
<i>steels, up to</i>		
0.45%	0.021%	0.015%
0.46-1.0%	0.048%	0.025%
<i>Alloy Steels :—</i>		
<i>Cast Iron, up to</i>		
0.7%	0.050%	0.030%

It is hoped that the above will be of assistance to those who may be called upon to make decisions in matters which relate to the homogeneity or otherwise of steel castings or forgings, and will lead to a just appreciation of some of the factors involved in the production and examination of these materials.

* *The percentages of variation found and suggested for composition are (as in the case of weight and length) percentages of the whole 100 units, not of the total of the particular element determined only.*