XXIII. METALLURGY

I.G. Experiences with Hydrogen-Resistant Steels

The following information was obtained at Leuna from Dr.Wyszomirski and Dir.Strombeck. For the early 250 ats hydrogenation stalls a 6% chromium steel (N6) was normally used. This had satisfactory hydrogen-resistance properties but its creep strength limited its use to pressures not exceeding 200 ats at 560°C. For 350 ats stalls 1% molybdenum and 1% tungsten was added to N6 to give a steel which was designated N7. These additions improved the creep properties but resulted in difficulties in fabrication and in heat treatment. Molybdenum and tungsten contents were accordingly reduced to ½% (N8) and the resulting steel was found to be perfectly satisfactory as regards heat treatment which simply consisted of quenching from 700°C. No annealing was necessary and no heat treatment was required after welding. The steel was suitable for vessels operating at 350 ats pressure and at temperatures up to 550°C. It could also be used for the cooler parts of 700 ats stalls.

An improved material was nevertheless required for 700 ats operation, particularly in the preheater elements. V2A could not be obtained because of the short supply of nickel and a new steel (N10) was developed which had the following analysis:-

Carbon	0.18 - 0.22%
Chromium	3.0 - 3.6%
Molybdenum	0.5 - 1.2%
Tungsten	$>$ 0.3%
Vanadium	$>$ 0.75%

Dr.Wyszomirski explained the theory behind the development of this steel. Pure iron-chromium alloys have a very poor creep resistance but this can be overcome if the steel contains <u>finely divided</u> carbides. In order to obtain a suitable <u>chromium/carbon steel</u> the chromium content must be limited. Too much of this component tends to absorb the carbides and also to increase the grain size of disseminated carbides thereby reducing creep resistance.

The composition of N10 is very critical. A minimum of 0.18% carbon is required to give the creep quality but a content of 0.22% must not be exceeded if welding difficulties are to be avoided. At least 3% of chromium is needed to give the necessary hydrogen resistance; more than 3.6 of this component has an adverse effect on creep properties. At least 0.5% molybdenum is required to give a workable steel but the 1.2% has the same effect as excess chromium in reducing creep resistance. Vanadium is necessary from creep considerations but this component appears to be responsible for much of the difficulty which has been experienced in getting an even distribution of fine carbide grains in the steel.

In addition to careful control of the composition of N10 the I.G. pay great care to its heat treatment. It is first heated to 1,050°C and, when it has assumed an even temperature, it is cooled through the range 800-600°C at a minimum rate of 25°C/minute. This is to avoid the complete transformation of austenite via martensite to pearlite. If this treatment does not result in even properties of the steel it is reheated to 1,150°C and the process is repeated.

A special heat treatment bench was developed dealing with preheater elements. A complete element was laid horizontally on a brick bench which was so designed that each leg of the preheater was immediately above a long 25 mm wide slot which extended the full length of the element. Compressed air could be injected through the slots by means of a series of jets. The element was heated electrically by passage of a direct current and even heating was ensured by providing a removable insulated cover fitted with auxiliary electric heating elements.

When the preheater element had attained a temperature of 1,050°C the top cover was removed and replaced by an arrangement by which air could be impinged on the preheater tube from above. During this change of covers the preheater element usually cooled to 850-900°C. Rapid control cooling down to 600°C was then carried out by means of the air jets.

Trials had been made with water for rapid cooling but this method was found to be less reliable than the air cooling method particularly in the case of fin tubes which scaled badly under water treatment.

Finally the steel was given a secondary heat treatment consisting of reheating to 680°C followed by slow cooling in air.

The I.G. claim that the above method of heat treatment results in at least a 33% higher creep strength at 560°C than is obtainable by oil quenching.

Enamelling of Preheater Bends.

It has been mentioned in an earlier section of this report that troubles had been experienced at Leuna with the erosion of preheater bends when working at high paste rates. In an attempt to overcome this erosion, experiments have been made with enamelled preheater bends. Difficulty was experienced in getting an even distribution of enamel powder over the inner surface of the tube and in cooling the bends sufficiently rapidly to avoid an uneven enamel layer resulting from the flow of molten enamel. Enamel is in any case useless for pressures above 325 ats because differences in expansion in enamel and steel led to cracking of the enamel layer.

Chromium-Plated Injector Rams.

Normal deliveries of hard plated chromium rams were found to be very satisfactory for pressures up to 50 ats, reasonably good for working up to 250 ats but useless for 700 ats pressure. The makers tried to improve the chromium surface by high frequency electric treatment but little success was achieved.

Material for Butene Dehydrogenation Reactor Tubes.

FF30, a 30% chromium steel with about 40% nickel and about 0.10% of carbon, was found to be satisfactory mechanically but it catalysed carbon formation in the reaction, particularly at low space velocity.

Material for Catalytic Cracking Regenerators.

For the regenerator of the catalytic cracking unit which was to be erected in the Knokuk plant at Niedersachswerfen the I.G. proposed to use a steel of the following composition:-

Chromium	0.8 - 1.0%
Silicon	0.5%
Carbon	0.12%

This steel was stated to have excellent resistance to oxidation.