The Tendency in Metallurgical Science and Technology in France —The Functions of IRSID and ATS—

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I. Preface

I am deeply honored to have been invited by your Institute to give the 3rd Yukawa Memorial Lecture before one of the largest and most competent audiences a steelmaker could expect, and this great honor is also shared by ATS and the French steel industry.

What is remarkable in Mr. Masao Yukawa's 40-year career is not only his original ideas on the siting and structure of modern gigantic coastal steel plants, and the model set for the world steel industry by his achievements, but also the wide field to which he extended his activity. He took a particular interest in the training of young engineers, and he believed in the power of technological progress and in the importance of exchanges with foreign countries.

For these reasons, I hope to serve Mr. Yukawa's memory, modestly but sincerely, in presenting this lecture dealing with metallurgical science and technology in France.

This subject is indeed a very broad and ambitious one and it appears quite difficult to present a complete and detailed description of this field in the time available. To simplify, we suggest limiting the scope of this lecture to ferrous metallurgy—iron and steel—although a significant part of what will be described could also be applied to the non-ferrous industry.

A mere list of the research programs of the different French laboratories working on ferrous metal-

lurgy would be long and tedious and so we propose:

- 1) in a first part to deal with the French scientific and technical educational system, the types of R & D centers and the functions of IRSID and ATS, and
- 2) in a second part to spend more time describing a few typical examples of research carried out by some of these laboratories.

II. French Educational System and R & D

1. Educational System for Engineers and Researchers

The educational system for engineers and researchers has a very specific feature in France as it consists of two quite different routes: either the "Grandes Ecoles", *i.e.*, Higher Colleges of Engineering or the universities.

It would certainly be outside of the scope of this paper to go into the details of this complex system with its various possibilities, and we shall limit ourselves to a brief—and consequently somewhat incomplete—view of it.

For many years and up to about 20 years ago the Higher Colleges of Engineering, the Grandes Ecoles (Ecole Polytechnique, Ecoles des Mines, Ecole Centrale des Arts & Manufactures, Ecole des Ponts & Chaussées, Ecole des Arts & Métiers, Ecole Supérieure d'Electricité, . . . to name only a few of them), were producing most of the engineers for industry and applied research, whereas the scientific universities were forming mostly professors and staff for fundamental research laboratories.

The structure has changed significantly within the last two decades: indeed the two routes remain distinct but they have been better adapted to the needs of industry and bridges have been established between them.

This "bipolar" system starts just after the French baccalaureat, *i.e.*, at the age of 18.

(1) For the *university route*, there is no entrance examination.

After the 1st two-year cycle and 2nd two-year cycle, the student obtains a "Maître ès Science" degree in a scientific field: Mathematics, Physics, Chemistry, Biology, . . . if he has passed the successive examinations. The student is 22 years old.

The 3rd cycle starts with a one-year specialization, for example in Metallurgy, leading to a Diploma called "Diplôme d'Etudes Approfondies" (DEA), or for some universities a "Diplôme d'Etudes Indus-

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trielles Approfondies" more industry-oriented. He can then spend 2 years preparing for a 3rd cycle thesis. He is 25 years old.

A limited number stay 2 or 3 years more to obtain a Diploma of Doctor of Sciences, which gives the possibility of promotion as a University Assistant Professor, but the vacancies are very limited.

(2) For the *Grandes Ecoles route*, the student after 2 to 3 years' preparation mostly based on mathematics and physics takes the entrance examinations which are very competitive: for the most difficult colleges 1 candidate out of 60 (or more) passes.

If admitted he obtains an Engineering Diploma after 3 years—at the age of 23 or 24—.

Three possibilities are then offered to him:

(1) most of them start their career, for example in industry,

(2) some of them, more research-oriented, can prepare a Doctorate in Engineering in 2 or 3 years, or

(3) they can enter the University 3rd cycle previously mentioned.

2. Metallurgical Research and Development in France

This brief introduction on the French educational system helps to understand better who is doing Research and Development in France, and where and how it is done.

In fact Research and Development in ferrous metallurgy are carried out in a large number of centers:

- in laboratories of the *Grandes Ecoles*
- in University laboratories
- in centers belonging to the steel industry proper
- in laboratories of large public service companies such as Electricity of France (EDF), Atomic Energy Agency (CEA), *etc.* . . .
- in laboratories of industries connected with the steel industry, either corporate centers such as the Technical Center of the Mechanical Industries (CETIM), or centers belonging to Companies (Vallourec, Pont-à-Mousson, Air Liquide, Renault, *etc.*)

For fundamental research, the CNRS (National Scientific Research Center) plays an important role of coordination between fundamental laboratories.

We shall also mention the financial help from the DGRST (Governmental Agency for Scientific and Technical Research) and from ANVAR, a governmental agency for aiding the practical application of research results or propositions in the different branches of industry.

Most of the large universities have one or several metallurgical or materials sciences departments (Paris Sud-Orsay, Grenoble, Nancy, Metz, Lille, Marseille, *etc.*).

If we accept the broad classification between process metallurgy and physical metallurgy it can be said that the university laboratories are dealing more with physical metallurgy.

For example, Paris Sud-Orsay University is well known in the French steel industry for its metallurgical research, in particular on the effect of hydrogen, an example of which will be given later.

A few universities are carrying out research connected with process metallurgy: the best known is probably Grenoble University where several groups are dealing with high temperature physicochemistry. This University in cooperation with IRSID's physicochemistry department has also played a leading part in "Thermodata", a world-known bank of critically evaluated data covering metallurgical thermodynamics.

As regards the *Grandes Ecoles*, 20 years ago, only a few of them had metallurgical research departments: they were generally small but some became famous: for example at Ecole Centrale with Prof. Portevin or at the Mining School of Paris with Prof. Crussard, to take only two examples.

However, there was a general need in France to set up extensive research in the *Grandes Ecoles* as it became obvious that scientific and technological training had to be backed up by sufficiently developed research laboratories. This expansion of research started at the beginning of the 60's and as a demonstration of this spectacular development we shall take the example of the Mining School of Paris.

When it was decided to meet this challenge of research expansion, three new laboratories were built outside Paris

- one in Fontainebleau, 70 km South East of Paris,
- one in Corbeil, 40 km South of Paris,
- one in Sophia Antipolis in the South of France,

some research activities remaining at the school. A foundation-like organization called ARMINES was founded (1967).

These laboratories cover a large field of research activities: Earth Science and Mining Engineering, Chemical Engineering, Materials Sciences, Applied Mathematics and Social Sciences.

For *Materials Sciences*, 3 centers are directly involved:

1. The "Materials Center" in Corbeil (100 Persons)

The main subjects can be summarized as follows:

- 1) Solidification in connection with the formation of internal defects (in particular an original approach to a quantitative analysis of the semi-liquid-semi-solid zone morphology during solidification)
- 2) Relationships between microstructure and mechanical properties, plastic deformation investigated under static and dynamic conditions
- 3) Ductile and brittle fracture mechanisms from microscopic and macroscopic points of view
- 4) Ceramic and fiber-reinforced composite materials.

2. The "Forming Center" in Sophia Antipolis (48 Persons)

Its activity focused on the material forming processes includes:

- 1) improvement of plasticity modelization, more accurate prediction of metal flow during forming, roll pass design, hot shearing, *etc.*
- 2) thermomechanical study of tool-metal contacts: friction, abrasion, *etc.*, and
- 3) lubrication during cold forming: rolling, draw-

ing, etc.

3. The "Mechanics of Solids" Center at Ecole Polytechnique (63 Persons)

The main activity of this center deals with:

- 1) understanding, prediction and modelization of elastic, elastoplastic and viscoplastic behavior of materials and structures, including very big structures such as nuclear reactors and off-shore platforms, and
- 2) special mention can be made of the model-testing of large structures subject to earthquakes, a type of study with which you are very familiar in Japan.

For teaching and research together, the total number of people at the Mining School of Paris in 1982 is 702, divided into 444 with scientific training and 258 technicians and clerks.

3. The Function of IRSID

The research centers belonging to the French steel industry (27 million t steel in the peak year of 1974, and 21 million t in 1981) can be classified in two categories:

- 1) the centers of the companies or steel plants: Usinor, Sollac-Sacilor, Ugine, Creusot-Loire, Imphy, Pompey, Aubert & Duval, etc.,
- 2) the IRSID corporate laboratory.

IRSID was founded just after World War II in 1946, when the French steel industry was starting to recover from the war and production was shared between a number of small companies.

The success of IRSID, its good start and rapid growth, are largely due to the merit and resolution of Mr. H. Malcor, IRSID's first Chairman.

The first laboratories were built at the end of the 40's at Saint-Germain-en-Laye in the suburbs of Paris.

But it soon became obvious to Mr. Allard, IRSID's President, that there was a need for a center for pilot research and development on processes, which could not be built at Saint-Germain-en-Laye, noise and red smoke not being compatible with the environment of this former home of the kings of France! Thus at the end of the 50's this pilot research center was built at Maizières-lès-Metz in Lorraine where most of the French steel was produced at that time. It was provided with fairly large-scale pilot facilities, for example a 6 t converter, an electric arc furnace of the same tonnage and some world-known developments were achieved there during the 60's and later.

Consequently IRSID has 2 large R & D centers, totaling 550 people:

- one at Saint-Germain-en-Laye near Paris: 10 900 m² on a 9 ha site (Fig. 1)
- one at Maizières-lès-Metz (in the east of France): 15 200 m² on a 23 ha site (Fig. 2)

Their programs are interconnected but, generally speaking, one can say that the Saint-Germain laboratories are oriented towards metallurgical aspects of the processes and steel properties, whereas Maizières-lès-Metz deals more with technological aspects of processes.

In the field of steelmaking, the goals of the research

Fig. 1. IRSID St-Germain-en-Laye.

Fig. 2. IRSID Maizières-lès-Metz.

work done in Maizières are:

- to improve knowledge in the field of the metallurgical phenomena related to the production of pig-iron and steel,
- to better control the different operations of the equipment by developing means for the measure, control and automation of the processes in order to set up continuous ones,
- to search for new processes.

Electromagnetic stirring within the continuous casting mould (called Magnetogyr process) and stirring the liquid metal by blowing gas through the bottom (LBE process) are among the many studies that yielded industrial applications and that will be discussed in more details later on.

The research work done in Saint-Germain-en-Laye concerns steel properties, the development of methods for investigation and control, and also ways of obtaining steels fitting closely the requirements of steel users. Main scientific topics are:

- Hot and cold rolling,
- Heat treatment,
- Steel properties (welding, fatigue, fracture, cold forging),
- Surfaces (oxidation, corrosion, coating),
- Chemical analysis,
- Non-destructive testing.

Figure 3 represents the relative proportions of the different activities for IRSID as a whole.

- A: Process (metallurgical and technological aspects)
 B: Steel properties
 C: Information, training
 D: Measurement, control, non-destructive testing

Fig. 3. Activity breakdown. (1982)

At this stage two remarks can be made:

(1) IRSID has facilities to tackle a problem on different scales:

- on a laboratory scale with sophisticated equipment which might be too expensive to buy or run in a laboratory belonging to a plant,
- on a pilot scale, with good-sized pilot units (converter, furnaces, rolling mills, . . .),
- on a full industrial scale in plant in cooperation with local teams.

IRSID's staffs, particularly from Maizières, spend a good part of their time in French steel works.

An example of the efficiency of such multiple-scale research is mixed blowing in LD converter with its 3 stages:

- 1) physicochemistry of disequilibrium slag-metal in LD blowing,
- 2) pilot tests on the 6 t converter,
- 3) industrial application on a large unit (60 t) at the Valenciennes works in cooperation with the steel plant engineers.

(2) The successive oil shocks have severely hurt a European steel industry not well protected against low price imports and unable to define a common policy. The French steel industry has not been spared by these crises and consequently the different companies had to merge one after another leading to the present situation in which practically all steel production comes from 2 groups, Usinor and Sacilor, financially state-controlled.

Each group has had to revise its research policy within its own structure and in relation to IRSID.

Despite the crisis, the French steel industry has made the decision:

- to maintain IRSID at its present size,
- to increase its total spending on R & D.

A close connection has been established but there is no general rule as to how the work is divided. In a very general manner one could say that the process up to casting and the measurement would be more IRSID's function, whereas each company would develop its own R & D research for products, often with the help of IRSID.

4. The Function of ATS

The Technical Association of the French Steel Industry (ATS) was founded, like IRSID, at the end of the war. Its task is defined as: "the promotion of technical development in the French steel industry".

Following the restructuration which took place in 1977, the members of ATS are the French steel works. Each works delegates its representatives—essentially engineers—to the different activities led by ATS.

As will be seen later, representatives of foreign steel companies can also participate in some of these activities.

ATS's main functions are technical promotion and coordination. A staff of 20 persons—11 high-rank engineers, at least of chief engineer level, and 9 secretaries—takes care of these technical functions, and all the general services (printing, mailing, book keeping, maintenance, . . .) are accomplished by the personnel of the French Steel Federation in the same building.

Among the various activities of ATS, the main one involves the technical committees, an activity somewhat similar to the ISIJ committees.

For each main production process or service, a committee has been created: a total of 20 ATS committees as indicated in the list below:

Coke ovens,	Metallurgy study
Blast furnace,	(special steels),
Oxygen converter,	Testing,
Electric arc furnace,	Analysis,
Continuous casting,	Maintenance,
Hot rolling,	Transport and Handling,
Cold rolling,	Data processing,
Energy,	Environment,
Measurement,	Industrial Medicine,
Refractory,	Technics of concertation
Metallurgy study	and QC circles.
(ordinary steels),	

They usually meet 3 times a year for 2 days either in Paris or at a plant. Normally 25 to 45 people attend, which is a satisfactory number for efficient work.

Each committee has a chairman appointed for 3 years. The appointment is renewable.

An interesting feature is the "Select Committee" which assists each chairman. It consists of about 8 senior representatives from other works, including one ATS and one IRSID representative, and helps the Chairman to draw up its programs and to discuss the efficiency of the work done.

The systematic attendance of a high-ranking IRSID member, department head or director, proved to be very helpful for a rapid dissemination of IRSID's research results in the different French steel works.

In order to go more deeply into the problems, each committee includes subcommittees which are permanent, and working groups which are temporary. The latter are usually made up of 4 to 8 specialists from different plants meeting 3 to 10 times within about 1 year before presenting their final report.

A total of more than 1 200 engineers from the

French steel industries are directly associated with these ATS committees, subcommittees and working groups.

Quite frequently representatives from outside are invited: University professors, R & D specialists from other industries, equipment manufacturers, suppliers, customers, *etc.* and they also play an active part in different groups.

Another interesting feature concerning the ATS committees is the participation of engineers from foreign steel companies.

The important committees invite foreign colleagues of the same field once a year to a 2-day meeting with a common program prepared by ATS. In the various committees, we thus have representatives from Austria, Belgium, Germany, Great Britain, the Netherlands, Italy, Luxembourg, Spain, Portugal.

They are European countries for reasons of distance but if any Japanese companies are ever interested in sending representatives from time to time, such a possibility would certainly be welcomed.

In the same line, every December, ATS organizes its annual conference which is open to foreign participants, French and English being the official languages with simultaneous translation. This 2-day conference is attended by more than 600 participants. Half the 40 papers come from France and among the 50 % from foreign authors we are very honoured to have 20 % of Japanese papers.

The technical cooperation inside ATS is recognized as a factor of progress for the French steel industry and two brief illustrations are given below.

(1) For example in the blast furnace committee the meetings include specialists from different works and different functions—men from the floor, from technical sections, from maintenance and from various research centers (steel companies, IRSID, Grandes Ecoles, Universities). The participation of people from such varying backgrounds is the source of a great deal of useful new information and results, on blast furnace theory, modelization, measuring equipment, technological devices, burden quality, refractory behavior, *etc.*

It also creates an atmosphere favorable to technical progress.

(2) For the same reasons, the working groups prove to be an efficient means of technical progress. As an example, after IRSID's LBE process was first industrially started at the Valenciennes works, ATS set up a working group in which all the French steel works using the LD process were represented, by production and development men, and there were also several participants from IRSID. They met every 2 or 3 months to put in common their practical experiences and difficulties and this free and complete exchange was a decisive factor in the rapid development of the LBE process in France as indicated:

French LD industrially equipped in LBE:

end of 1980: 8 out of the 17 LD

end of 1981: 15 out of the 17 LD.

Many other examples showing the fruitful effect on technical progress of this interchange of results for

nonconfidential subjects in the ATS meetings could be quoted.

III. Some Examples of R & D

1. Blast Furnace Theory—Its Application to Low Fuel Rate (*Ecole Centrale de Paris*)

At Ecole Centrale, Prof. Rist is conducting a research program on blast furnaces theory: optimum burden reduction, softening of sinter, mechanical forces inside the shaft, *etc.*

When Prof. Rist was at IRSID, he proposed the "operating line" (Fig. 4), which summarizes the oxygen equilibria of an isothermal reduction in the blast furnace, by means of a single straight line.

The slope of this line is a measure of the fuel consumption. When the line PA touches on the W corner, the reduction is ideal and the fuel rate is the lowest possible.

When the line moves away from W , reduction becomes non-ideal, characterized by the ω coefficient, and the fuel rate increases.

We shall give two new developments showing the usefulness of the operating line and its ω coefficient.

(1) In blast furnace practice much progress has been achieved from the "central peak temperature" technique initiated and developed in Japan (Fig. 5), as it offers great advantages: higher productivity, lower heat losses, longer life of refractory and better Zn removal.

But reduction conditions are quite different in the center where the fusion zone is high and residence time is short, and near the wall where it is the opposite.

How does this peak influence the total fuel consumption of the blast furnace?

Prof. Rist uses a schematic description of the blast furnace where the shaft is considered as the sum of three concentric shafts, at the wall, mid-radius and at the center (Fig. 6). For each elementary shaft it is

Fig. 4. Rist's operating line.

possible to draw an operating line illustrating the O and C balances.

The increase in permeability in the center can be obtained in different ways: for example by a higher coke/sinter ratio or by a coarser burden.

In the first case (1) with a higher coke/sinter ratio, the gas encounters but a small flow of sinter and its oxidation degree remains low. Reduction is not ideal in this zone as shown in Fig. 7(1).

On the other hand in the second case (2) with coarser burden in the center, reduction conditions improve through a higher flow of sinter to meet the gas and a better gas oxidation (Fig. 7(2)).

This second solution corresponds to a significantly lower coke consumption.

This has been confirmed industrially on the Solmer blast furnace, which held the world record for low fuel consumption—443 kg/t iron in an all-coke practice—last year. This corresponds to the ideal reduction of burden in the shaft for isothermal conditions.

(2) But even lower fuel consumption is still achievable as Prof. Rist has recently shown.

In the “peak temperature technique” the sinter charged in the peripheral zone near the wall has a long residence time and a slow temperature gradient. Under such conditions and with a sinter of high

reducibility, the reduction in the shaft can follow a path more favorable than the isothermal one, as shown in Fig. 8.

The graphical representation shows that in extreme conditions the reducing gas in the shaft can reach 2 equilibria successively (with wustite W' and with magnetite M'). Compared to its isothermal PE position, the line shows a negative ω coefficient, as indicated in Fig. 8.

As the slope of the line gives the fuel consumption, one can see that the new path corresponds to a lower fuel consumption, the difference being 60 kg of coke

Fig. 5. Central peak T .

Fig. 6. Concentric shafts.

Fig. 7. Shaft reduction.

Fig. 8. Rist's reduction in T gradient.

per tonne of iron.

This reduction in a temperature gradient, and the corresponding research program of producing sinter of high reducibility to take full benefit of it, opens a new challenge for the blast furnace men and Solmer has already achieved a first step in that direction with an all-coke consumption of 439 kg of coke per tonne of iron in May 1982, corresponding to a negative ω coefficient.

2. Electromagnetic Stirring (EMS) in Continuous Casting (IRSID Maizières)

It has been known for many years that coils connected to an alternating current could in certain conditions induce a stirring action inside a liquid pool of steel. But it was not until the introduction of the continuous casting process that this technique of electromagnetic stirring (EMS) found practical applications.

IRSID has been among the pioneers in this field and has played an important part in its industrial development.

At the beginning, magnetic stirring was applied to the secondary cooling zone of billet and bloom casters (Fig. 9).

This technique prevents the formation of minigots near the tip of the liquid pool and its beneficial effect on axial segregation and porosity has been confirmed on many continuous casting machines.

The French company SAFE was the first steel mill in the world to install EMS on the 4 strands of its caster in 1973 for systematic industrial use.

EMS in the secondary cooling zone was then applied to slab casting (Fig. 9) where it brought significant improvement in internal quality, especially for plates.

But EMS applied to the secondary cooling zone cannot have any action on the already solidified shell, hence the interest of stirring as early as possible—i.e., in the mold itself—in order to affect the surface and subsurface zones.

The problem is technologically more difficult because space is limited and the copper mold acts as a shield for the magnetic field and a lower-frequency generator must be used.

Linear stirring in the mold was the first technique experimented by IRSID at Allevard in the early 70's but the real breakthrough was achieved when IRSID and Usinor in cooperation with Arbed experimented with rotary stirring in the mold. This technique, called Magnetogyr, has since been developed into a successful process applied to square and round sections of the entire range of bloom and billet.

Figure 10 shows the principle of the process with the induction coil inserted inside the water cooling chamber of the mold. The rotation creates a deep meniscus at the bottom of which non metallic inclusions accumulate and can be removed. They are thus prevented from being entrapped in the skin when it first solidifies.

The improvement in the outer surface quality is spectacular when pouring out with an open stream. The number of slag entrapments (Fig. 11) is virtually reduced to zero and the number of pinholes becomes extremely small. Surface conditioning can be greatly reduced or eliminated, meaning a significant money saving. The result holds true for submerged nozzle casting although the difference is not so spectacular since casting with powder guarantees a good surface quality.

Similarly Fig. 12 shows the spectacular improvement in subsurface quality for open stream casts in a curved CC machine.

For slabs, stirring in the mold was started by IRSID (NETINEL process) in 1978 on the Dillingen slab caster, showing a significant improvement in the subsurface quality of low C Al killed sheets. Work on this NETINEL process is continuing.

The magneto-hydrodynamic flow created by EMS is composed of one or several loops. Examination of the macrostructure of stirred samples, water or mercury modelization and theoretical calculation are providing some information on these vortices and their velocity, which can vary in the range of 0.1 to 1 m/s, but more has to be known quantitatively on these flow patterns.

Fig. 9. Applications of EMS.

Fig. 10. Metallurgical principles of the magnetogyr process.

Fig. 11. Comparison of slag entrapments.

Fig. 12. Subsurface cleanliness of 135 mm round billets.

French universities in conjunction with IRSID are studying different approaches to mathematical modelling of electromagnetic stirring in continuous casting. Grenoble University has carried out hydrodynamic calculations for rotary stirring of round billets, tackling the difficult problem of a finite-length inductor. The Mining School of Nancy and IRSID have worked out a model of crystal multiplication and growth which seems to be the first attempt to model the formation of the equiaxe zone. Figure 13 shows some of the first results obtained recently on the local solidified fractions and superheat for different mixing conditions.

Fig. 13. Equiaxe zone model.

3. Direct Quenching of Plates—RAC—(Usinor)

For several years the demand has been growing for steels of high tensile and fracture-toughness characteristics and being suitable for welding, for various industrial applications such as large-diameter welded pipes, offshore platforms, bridges, tanks, shipbuilding, etc.

The controlled rolling technique has already achieved great progress in this respect, but it approaches its limit for grades X 80 strength and for

thicknesses of 25~30 mm. Beyond that the conventional solution is to carry out a separate quenching treatment after having reaustenized the plate.

USINOR has chosen another route, direct on-line quenching, which avoids the drawbacks of a separate quenching treatment and offers important metallurgical advantages described later.

For this development, USINOR cooperated with the BERTIN Cy, a French company well known for its aero- and hydrodynamic achievements, and with IRSID.

1. Description of RAC Machine

The highest quenching rate obtainable is achieved with a "sheet" of water in forced motion on the plate surface.

This principle is applied in the RAC (controlled accelerated cooling) machine. The plate moving inside a tunnel is cooled down by a water stream circulation from one end to the other, in channels defined by the tunnel walls and the plate (Fig. 14).

The present industrial prototype (Fig. 15) installed behind the hot leveller of the Dunkirk plate mill in 1978 is built for plates 8 to 50 mm thick and 12 to 16 m long but its modular-type design makes it possible to deal with longer plates by adding complementary units.

2. Metallurgical and Technological Results

(1) Direct quenching takes full advantage of elements such as niobium, which would not fully return into solution during the reaustenizing treatment preceding a conventional quenching.

(2) The average cooling rates from 800 to 500 °C achieved at the plate core vary from 120 °C/s for plates 10 mm thick to 30 °C/s for those 30 mm thick.

Fig. 14. Principle of the RAC process.

Fig. 15. RAC industrial prototype. (USINOR Dunkirk)

These values are the highest obtainable with water quenching.

(3) The uniform quenching confers a good homogeneity of metallurgical properties inside the plate: for example for a plate 10 mm thick, 13 m long and 2.5 m wide, the hardness profile in the core along the axis does not vary by more than 50 points of HV 30 (for a level of 380), except for the ends which are cut off.

(4) The as-quenched plates have excellent flatness due to the uniform quenching.

(5) For an identical equivalent C, directly quenched and tempered plates obtained on the RAC machine show yield strengths higher than those of the best plates obtained by controlled rolling and by normalizing as shown in Fig. 16. As an example, for an equivalent C of 0.40 %, the yield strength reaches a value of more than 690 N/mm² for 15~20 mm thick plates.

(6) With controlled rolling, the final austenite is work-hardened. It produces a refinement of the martensite structure after quenching, which gives an increase in the hardness at plate core, and also greatly improves the transition temperature.

For example, plates on 30 mm thick of the same composition passed through the RAC machine the result after tempering is:

{	TK 50 %:	-25 °C with conventional
	(rolling direction)	rolling ended at 990 °C
		-105 °C with controlled rolling ended at 860 °C

(7) The weldability of RAC treated plates is excellent due to the lower level of chemical composition necessary to provide the mechanical properties required, and numerous studies have been made on the weldability of these steels.

For example, Fig. 17 shows the composition and CCT diagram for 2 steels having the same guaranteed mechanical properties, including yield point ≥ 460 MPa.

The two sets of curves are significantly shifted on the 800 to 500 °C cooling scale. The time lengths are on average 8 times shorter for the RAC steel compared to the conventional ones.

Fig. 16. Yield strength.

Fig. 17. CCT diagram.

The mechanical tests on as-welded and stress-relieved assemblies have proved the excellent weldability and good properties in the heat-affected zone of the RAC steels.

4. A Theoretical Approach to Ironing of Cans (SOLLAC-SACILOR)

SOLLAC, one of the French producers of steel for drawn and ironed cans, is carrying out a research study on the influence of various parameters upon the stresses developed during the ironing operation of cans.

The stress calculations are based on the slice method with the following hypotheses:

- (1) The material has a very low strain-hardening coefficient.
- (2) It follows the TRESCA criterion with a flow limit σ_0 .
- (3) The friction forces are of the Coulomb type with coefficients μ_m at metal/die interface and μ_p at metal/punch interface.
- (4) The wall being thin, deformation remains two-dimensional.

It is outside the scope of this paper to go into the detailed mathematical development of this research study and we shall present only some results.

The equilibrium of a slice is represented in Fig. 18(a).

Calculation shows that the equilibrium equation is:

$$\sigma_x dz + z d\sigma_x + P(1+A)dz = 0$$

with
$$A = \frac{\mu_m - \mu_p}{\tan \alpha}.$$

The ironing stresses shown in Fig. 18(b) are obtained by the following equations:

Fig. 18. (a) Stress definition and (b) Geometry.

1st case $\mu_m \neq \mu_p$
$$\frac{\sigma}{\sigma_0} = \frac{1+A}{A} \left[1 - \left(\frac{h}{h_0} \right)^A \right]$$

2nd case $\mu_m = \mu_p$
$$\frac{\sigma}{\sigma_0} = -\text{Log} \frac{h}{h_0}$$

From these equations, the influence of the main parameters on the ironing stresses in the drawing gap can be deduced.

It can also be noticed that with the plasticity criteria retained, i.e., $p + \sigma_x = \sigma_0$ pressure variations during ironing can be deduced by symmetry from stress variations.

Figure 19 shows the variation of the ironing stress as a function of the reduction ratio $\tau = (h_0 - h)/h_0$ for different values of A, i.e., of α and $\mu_m - \mu_p$.

From this figure it can be observed that:

- (1) When the reduction ratio increases, the ironing stress σ increases.
- (2) When $\mu_m - \mu_p$, the stress variations become independent of the α die angle.
- (3) For a given reduction ratio τ and a given $\mu_m - \mu_p$, if $\mu_m - \mu_p > 0$ the stress decreases when α increases if $\mu_m - \mu_p < 0$ the stress increases with α .
- (4) The smaller the α angle, the more sensitive σ becomes to small variations of $\mu_m - \mu_p$.
- (5) For low values of α ($\approx 5^\circ$); and low negative values of $\mu_m - \mu_p$ the stress approaches zero and can become negative, that is to say corresponding to extrusion. In such cases, the transition from tension to compression is very sensitive to small variations in the friction coefficients.

The maximum reduction ratio can also be deduced:

if $A \neq 0$
$$\tau_{\max} = 1 - \left(\frac{1}{1+A} \right)^4$$

if $A = 0$
$$\tau_{\max} = 1 - \frac{1}{e}$$

It can be shown that:

- (1) an increase of friction on the punch side compared to the die side, leads to high reduction ratios, especially with low values of the α angle, and
- (2) the lower the α angle, the more sensitive the

Fig. 19. Stress vs. ratio curves.

reduction ratio will be to variations of friction conditions and this could lead to unstable ironing conditions.

In a second part of this study, the forces developed on the punch and on the die are calculated and similarly the influence of the ironing parameters is deduced.

The next part of this research will consist of determining the ironing behavior of different black plates with various lubricants, and from the values of the forces, of calculating the real friction coefficients in order to put them back into the mathematical model developed.

But already at this stage, this research appears to be quite a useful tool for a better understanding of the phenomena occurring during the ironing process.

5. Hydrogen Embrittlement of Steel (Various Laboratories Including Orsay University)

The mechanisms of steel embrittlement by hydrogen are multiple and complex, and a large number of laboratories are carrying out research programs and cooperation in this field. They include Universities (Orsay and also Compiègne, Nancy, Valenciennes), the Ecole Centrale, the Electricity of France (EDF) and Gas of France (GDF), Atomic Energy (CEA), Air Liquide, IRSID, the Institute of Welding, and most of the steel companies (Creusot-Loire, Pompey, Imphy, Usinor, Sollac-Sacilor, . . .).

As an example we shall summarize the work at the Hydrogen Department of Orsay University.

It concentrates on the influence of the microstructure of metals—and more particularly of industrial

steels—on their behavior in contact with hydrogen.

(1) The first step is to determine with accuracy the microstructure of the metal specimens by electron microscope of 100 or 1 000 kV (at CNRS, ONERA), or by more specific techniques, *e.g.*, Auger Spectrometry, IRSID's glow discharge spectroscopy.

(2) Then hydrogen is introduced by cathodic charging, either conventionally or in gaseous solutions (Air Liquide and CEA), or by a special technique using fused salts at 120~220 °C, particularly interesting for materials of low H₂ permeability such as the austenitic structures.

(3) During or after the introduction of hydrogen:—its mobility is determined by permeation, degassing or counting,

—its location can be directly observed with the 1 000 kV electron microscope by the autoradiography of tritium introduced in the same conditions as hydrogen (Figs. 20 and 21),

—microscopic damage can be directly observed with the 1 000 kV electron microscope on hydrogenated thin foils,

—macroscopic damage is determined by mechanical testing.

Examples of research on hydrogen at Orsay University:

(1) H₂ diffusion and trapping in Cr-Mo steels (with Air Liquide and IRSID).

It involves the relationships between the different martensitic microstructures obtained by quenching and tempering of 1%Cr-0.2%Mo steels and the trapping processes of H₂ observed by tritium autoradiography. The morphology and chemical com-

Fig. 20. Principle of autoradiography.

Fig. 21(a). H_2 trapping on dislocation. (grade 304 L)

Fig. 21(b). Tritium autoradiography. (grade 304 L)

position of the M_3C carbides, the nature and thickness of the oxide films and their barrier effects have been studied.

Autoradiography has shown different types of H_2 trapping of weak, medium or high energy according to the structure, and the behavior of a metal can be forecast according to the proportion and distribution of these different trappings.

(2) Behavior of weldable steels ($YS \geq 360 \text{ N/mm}^2$ grade) in an environment of H_2 and SH_2 (with Creusot-Loire, Ecole Centrale and IRSID).

It has been found that:

—the trapping sites of medium energy, such as manganese sulfides, have the most detrimental effect on cracking without external stress, while sites of high energy, such as cerium sulfides, are good trapping sites; and

—to explain delayed fracture and induced cracking, the influence of hydrogen transport by dislocations, of stress concentration and of notch sensibility must also be considered.

(3) Behavior of austenitic structures in an H_2 environment (with CEA, Gas of France and other universities).

It deals with stainless steels of 304 L and 316 L (stable) grade, with nickel and Inconel alloys.

The H_2 trapping phenomenon by dislocation has been displayed by autoradiography for the first time in these γ structures and an important phenomenon of accelerated transport by dislocations during plastic deformation has also been shown. This phenomenon is the cause of the loss of ductility observed in an H_2 environment (Fig. 21(a)).

Cathodic hydrogen at room temperature produces important superficial phenomena of induced martensite transformation. The sensitivity of these stainless steels to hydrogen cracking is directly connected to the γ structure stability: the existence in these steels of strain-induced martensite ϵ and α' is particularly detrimental. The unfavorable influence of some sensitization treatment has been demonstrated and explained.

Similarly important results have also been found with nickel and its alloys.

6. Application of Glow Discharge Spectroscopy to Surface Analysis (IRSID Saint-Germain)

The behavior of protective coatings, their adhesion and resistance to corrosion, depend greatly upon the physical and chemical properties of the surface layers. It is thus essential to carefully determine the chemical composition of the metal surface, not only for the end product but also during the various processing stages, by using a fast and accurate analytical method.

The conventional methods such as AES, SIMS or ESCA are long and complex.

A new method based on optical emission spectroscopy has been developed by IRSID.

The light source is a Grimm lamp of a special type (Fig. 22).

The electrical field (400 to 2 000 V) ionizes the argon carrier gas and the ions produced dislodge the surface atoms, layer by layer. Radiations characteristic of each element are emitted. The complete apparatus has been described in the literature.

In practice a few seconds of sputtering are sufficient for the scanning of a steel surface.

Figure 23 shows the surface profile of a steel sample after 8 min of sputtering (1 000 V, 100 mA).

(1) Layers $100 \mu\text{m}$ thick can be explored in 30 min, and by some special devices sputtering times of up to 3 hr can be achieved.

64 elements can be analyzed simultaneously and instantaneously.

For instantaneous analysis, the detection limit is 0.001 wt% (10 ppm) for metallic elements and metalloids such as C, S, P and B, and 0.1 wt% for elements such as H_2 , O_2 and N_2 .

For the quantitative analysis of metal and chemical

compounds for which a calibration can be made, good accuracy is achieved.

The relative error generally ranges between 0.2 % and 0.5 % and is in any case inferior to 1 %.

IRSID has been using this method for several years for the surface analysis of steel and some examples are given below.

(2) Scale analysis:

It is known that the composition of scale depends on the grade of steel and on the condition of formation.

Figure 24(A) shows the concentration profile for iron and oxygen in oxide scale formed in hot rolled steel.

Figure 24(B) shows how different the behavior of these elements can be in the scale itself and in the interface zone compared to the average content in the steel.

Fig. 22. Schematic diagram of Grimm glow discharge lamp. (IRSID)

Fig. 23. Surface profile.

Fig. 24. (A) Fe and O contents and (B) Other elements.

(3) Phosphate coating of mild steel sheets:

The analysis by glow discharge spectroscopy of a layer of zinc phosphate deposited on a sheet of mild steel as pretreatment before painting has shown how heterogeneous such a deposit is throughout its thickness.

(4) Passive film analysis:

The corrosion resistance of stainless steels is due to the existence of very thin layers of passivity (a few nanometers) found on the surface of the metal. Glow discharge spectroscopy is now in widespread use for surface characterization of such sheets.

Figure 25 gives an example of concentration profiles for a stainless steel of type 316 L which has undergone conventional polishing treatment. It clearly shows:

- a Cr enrichment in the oxide film of a few nanometers,
- an Ni enrichment near the metal oxide interface,
- the presence of elements such as Cu and Si in the layers.

In conclusion, by its simplicity and speed glow discharge spectroscopy developed by IRSID is a very attractive method for the inspection of steel surfaces

Fig. 25. Passive film—316 stainless steel. (electrolytical polish)

and for the development of new coatings. Combined with other conventional techniques it proves to be a very useful tool for scientific and technical progress in these fields.

7. Steels for Nuclear Power Stations (EDF, CEA, Creusot-Loire, Imphy, Ugine, Framatome, Novatome, etc.)

In the last two decades France has considerably developed its production of electricity of nuclear origin and in this new industry steels are used in constrained conditions and with high safety levels.

The following Fig. 26 shows the increase in production of electricity from nuclear power stations in France.

This development is scheduled to continue in coming years as indicated in Fig. 27 showing nuclear power plants, in production, under construction and officially planned, with the different types of reactors:—the old graphite-gas reactors,
—the pressurized water reactors (PWR 900 and 1 300 MW series),
—the fast breeder reactors.

Phenix 250 MW in operation

Superphenix 1 200 MW under construction due to start mid 1984

The components of nuclear power stations present some specific characteristics:—Their size and weight; for example the vessel dimensions of a 1 300 MW PWR reactor (Fig. 28) with its cover are:

height : 13 m

inside diameter: 4.40 m

wall thickness : 220~500 mm

weight : 420 t

—The number and severity of conditions to be simultaneous by met: mechanical characteristics (in cold and hot state, under different stress conditions), resistance to corrosion in severe conditions, structure stability under radiation, good formability, weldability, machinability.

—An excellent safety level.

—In industrial conditions of cost and delivery time.

Extensive R & D programs have been and are still being carried out in this fields by a number of French metallurgical laboratories. We shall briefly summarize three examples.

(1) In the boilers of PWR nuclear power stations, the primary cooling water circuits are working in a

Fig. 26. French nuclear production development.

Fig. 27. French nuclear power plants.

Fig. 28. 1300 MW nuclear plant vessel, Framatome-le Creusot.

temperature range of 290 to 350 °C. The materials must show good guarantees of structure stability in the temperature range and for periods of up to 40 years.

A part of the equipment is presently made of austenitic stainless steel but this type of steel has the disadvantage of a low yield strength (200 to 300 N/mm² at 20 °C).

On the contrary martensitic stainless steels, tempered at temperatures of 520 °C or more, show a much higher yield strength of 600 to 1200 N/mm² at 20 °C. But embrittlement has been observed in these grades of steel when maintained at temperatures of 520 °C or less.

The Imphy research center in cooperation with Framatome has studied the influence of different alloying elements on this embrittlement of martensitic stainless steels.

A series of heats were melted in the following range: very low C 0.03 %, Cr 12~16 %, Ni 0~8 %, Mo 0~2 % (and for two structural-hardening grades Cu 3 % or Al 1 %).

They were austenized for 1 hr at 1010 °C, oil-quenched, tempered at 590 °C then aged at 450 °C for between 10 hr and 10 000 hr.

Figure 29 confirms that these steels (numbered 3 to 11) harden compared to the austenitic reference steels (numbered 1 to 2).

It has been proved by sophisticated measurements that:

—as in ferritic stainless steels, the embrittlement results from the homogeneous precipitation of an α' Cr rich phase. This is clearly shown in the example of Fig. 30

—embrittlement increases with the Cr+Mo content

—embrittlement decreases when residual austenite increases in the tempered steels.

In conclusion, this research shows that embrittlement due to aging of martensitic stainless steels does

Fig. 29. Embrittlement phenomenon—martensitic stainless steel.

not present an insuperable handicap (see curves numbered 3 and 7 in Fig. 29) as long as the steelmaking process is carefully controlled and the composition is based on 13 % Cr and 4~8 % Nickel contents.

(2) In order to fulfill the need for a better yield from uranium, the fast breeder reactor technique is being developed in France (Fig. 31).

There are 3 stages in this development.

- 1) “Rapsodie”, a first experimental unit, not intended to be connected
- 2) “Phenix”, a 250 MW reactor in operation since 1973
- 3) “Superphenix”, a 1200 MW reactor under construction and due to start mid 1984.

The metallurgical problems faced are significantly more difficult than for the PWR power stations for at least 3 reasons:

(1) The components are even bigger: the main vessel of Superphenix has a height of 19 m and a diameter of 21 m.

(2) The temperature in the circuit tubes is higher, max. 550 °C compared to 390 °C max.

(3) Molten sodium is used as a coolant.

Only austenitic steels are used in the primary circuits of these reactors. But after long periods at elevated temperatures, a complex series of precipitation reactions occurs in these steels and it was necessary to investigate the consequence of these reactions on the structure and mechanical properties of these steels. The influence of the molten sodium medium on the mechanical properties also had to be investigated.

In order to meet this challenge an extensive research program has been launched by the laboratories associated in an “Electricity of France–Atomic Energy Agency Industry” Work Group and the Research Center of Creusot–Loire (Unieux), in cooperation with Novatome.

The results obtained have made it possible to define narrower ranges of steel compositions with more clearly-defined properties and quality.

This is summarized by the following chronological table:

Rapsodie: grade 316

Phenix: grade 316 L

Superphenix: SPH grade specification (%) C
0.030 max. Mn 1.6~2.0 Cr 17.0~18.0 Ni 12~
12.5 Mo 2.3~2.7 N₂ 0.06~0.08 B 0.0015~
0.0030

A similar SPP grade was developed by Imphy for forgings. The forging characteristics are the same as those of the SPH grade.

The choice of the chemical composition and the narrowness of specifications selected for this SPH grade provides satisfactory guarantees with:

- good creep behavior and low-cycle fatigue endurance in sodium medium
- satisfactory toughness defined as the remaining KIC_U impact strength after 100 000 hr at a max. T of 550 °C
- homogeneous Cr content throughout their work life, the steel least prone to intergranular sensitization phenomena
- good weldability, in particular concerning the risk of cracking in the heat-affected zone.

This narrowing of specifications implies that all the know-how of steelmakers is required to achieve this aim.

(3) Research was also undertaken to find austenitic grades suitable for the severe conditions encountered in nuclear fuel reprocessing plants. Special grades containing very low concentrations of residual elements were developed by Unieux Creusot-Loire laboratory in the 18/12 (304 L) and 18/14 families to withstand corrosion in a concentrated nitric acid medium.

IV. Conclusion

The brief review of the educational system and structure of metallurgical R & D in France, and the short description of IRSID and ATS have been followed by a few examples as illustrations.

These examples have been very diversified:

- from theoretical aspects to technological ones,
- from huge blast furnaces to small beer cans, and

—from 500 mm thick plates to 4 μ layers, a million times thinner.

This variety, which might appear to you somewhat confusing, gives however a good picture of the vitality of metallurgical research in France. And if some one from another industry had to deliver a similar paper on his speciality he would probably have the same large choice of examples at his disposal.

Research is vital for countries like France which are poor in natural resources, and this is the reason

Fig. 31. Fast breeder power plant. (superphenix)

Fig. 30. α' phase precipitation. (16 % Cr, 4 % Ni, 10 000 hr, 4 500 °C).

why the policy for years to come is to increase our research potential in industry, including the steel industry.

The objective announced recently by our government is to increase total R & D expenditure in France by 40 % by 1985, which is considerable.

But Research is not an end in itself and it must be followed by fully industrial applications, at least in a reasonable number of cases. Increasing attention is being drawn to the desirability of this, but it is not always an easy step to take, for several reasons, not all of them financial.

Several organizations in France are working towards this objective, and for the steel industry it has been shown that ATS and IRSID are contributing to the partial solution of this difficult problem.

It is however realistic to recognize that much still remains to be done in this field in France.

Japan is known to have solved this question of the transition from Research to fully industrial development much better, and we in France have a great deal to learn from you in this crucial matter.

Thank you very much for your kind attention.