

worked with or without oxygen, the protective oxide film which formed was the same. It was a magnetic iron oxide ( $\gamma$  - Fe<sub>2</sub>O<sub>3</sub>) formed at low temperatures, in contrast to a closely-related oxide ( $\alpha$  - Fe<sub>2</sub>O<sub>3</sub>), usually found only at high temperatures.

In subsequent allied experiments, NRC investigators demonstrated that an identical oxide film could be obtained electrochemically and also by reducing the impurities on the metal surface with hydrogen and then simply exposing the "cleansed" iron to the air at room temperature.

"Our metal world runs on this film of oxide," says Dr. Cohen. "Almost all metals are potentially flammable and if divided finely enough or brought to high enough temperatures will burst into flame and burn. Some ships have been turned into fiery infernos because of it. But the thin oxide film which protects against corrosion is what prevents such a phenomenon from happening. Formed in initial exposure of the metal to air, this film protects the metal from the environment. Only gold is stable without its oxide film, although gold too forms a very thin oxide layer, just a few atoms thick."

From these early experiments, NRC's work in the field of corrosion research has been considerably broadened in scope. The formation of metal oxide films at high temperatures has been one of the specialty areas. This work was actually begun in 1947 under Dr. Cohen whose particular interest initially was the formation at elevated temperatures of oxides of nickel, iron and chromium (the basic elements of the stainless steels) and their alloys. Here the emphasis is on both chemistry and metallurgy, supported by visual and electro-optical techniques and kinetics studies. The aim is to characterize the form and crystal structure of these oxides and to determine the reactions involved. This information is then related to elucidating the effect of such oxides on general oxidation behavior.

The research brought several important aspects of oxidation behavior to light. It demonstrated that the initial preparation of the surface has a marked effect on subsequent oxidation behavior. The adherence of the thin oxide film and hence its effectiveness as an inhibitor against further oxidation and resultant corrosion depends directly on the initial cold-working (deformation) of the metal and on the impurities in the surface. The studies also showed that if one of the metal components in an alloy is oxidized easily this sometimes leads to an increase in the overall rate of oxidation (as is the case when manganese is added to a chromium-iron alloy) and sometimes to a decrease in rate of oxidation (such as occurs with the addition of aluminum or chromium to an alloy).

This area of research is of critical importance. The efficiency of atomic energy and steam energy plants increases with elevated temperature — but so does corrosion. Break-downs in nuclear reactors and steam generators most frequently result from problems with materials. This means corrosion problems, specifically high-temperature corrosion problems.

Many techniques developed in the NRC Laboratory have proved of great value to industry and have gained wide acceptance. The NRC methods have enabled researchers to investigate the rates of formation and the physics and chemistry of films less than 40 billionths of an inch thick in atmospheres where both the temperatures and concentrations of reacting gases are closely controlled.

An important aspect of current research in this Section is directed at refining and exploiting an analytical technique for structural and chemical information about thin films on metal

crystals. This technique, which is proving to be increasingly useful to industry, combines high-energy electron diffraction with X-ray analysis procedures. It involves a focussed electron beam which strikes the specimen at a low angle and is diffracted from the surface layers of the crystal. The resulting electron diffraction patterns which appear on a fluorescent screen are affected by both the size and crystalline structure of the surface. But in addition to providing structural information through diffraction, electrons from the same beam furnish chemical information by the excitation of X-rays which are characteristic "fingerprints" of the chemical elements in the surface. The X-ray spectra serve to identify the elements present and the intensity of the X-ray emission indicates how plentiful these elements are at the crystal surface. The spectra are sensitive to the bonding of the atoms in the surface and studies have been initiated to use this sensitivity to gain a better understanding of the chemical bonding in the oxides. The NRC scientists are also investigating this chemical bonding in some of the oxides by means of a resonance technique (Mossbauer effect).

The Metallic Corrosion and Oxidation Section has solved corrosion problems involving hot water tanks and metals on experimental exposure sites for the benefit of NRC's Division of Building Research. As well, the Section has developed special techniques for preparing surfaces of metal staples to be used in suturing. This work was done for NRC's Division of Mechanical Engineering.

But a significant portion of the work of this Section involves helping industrial firms, giving advice on request, and at times undertaking laboratory studies on a very broad range of corrosion and corrosion-related problems encountered in manufacturing processes: corrosion in sulphur melters for paper mills, the staining of aluminum cast or machine parts in storage (which provides points for the initiation of stress corrosion), corrosion failure in aircraft parts and sections, corrosion in brass, problems with antifreeze and radiator corrosion, electro-optical examination of thin-films on metals for metal fabricators, problems in electropolishing metal surfaces of interest to a pharmaceutical firm — these and many more have been tackled and solved by the Section.

"We are one of the very few general corrosion labs in Canada, although there are other more specialized labs," says Dr. Cohen. "Our increased understanding of basic corrosion processes provides a solid foundation for the basic problem is understanding corrosion. Almost everything we have done in terms of gaining a better basic understanding eventually has become useful to somebody, often in quite a different area. Our research into the electrochemical behavior of magnesium, used for protecting ships and pipelines against corrosion, has attracted more attention from battery manufacturers than from corrosion engineers. Our techniques for studying thin films is receiving considerable attention from the thin-film electronics industry." □ Earl Maser

*View of the first high-temperature apparatus used by the Section. • Le premier appareil, de la section, pour températures élevées.*