Surface study on vacuum fired stainless steel by AFM and STM

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Stainless steel (SS) is one of the most commonly used constructional materials for vacuum chambers and components. Special applications like accelerator, storage ring facilities or advanced semiconductor device processing make need for extreme high vacuum (XHV). If the XHV is to be achieved a reduction of the outgassing rates of the materials used in the construction of the vacuum system is essential [1]. Beside surface treatment to reduce the surface roughness high temperature vacuum firing became an alternate method and widely accepted practice of reducing the amount of hydrogen dissolved in SS. For the description of the outgassing rate basically two models common as diffusion limited model (DLM) and recombination limited model (RLM) have been discussed [2-5]. Surface states which may greatly influence the outgassing kinetics are not considered in the DLM. The recombination rate of hydrogen atoms approaching the surface from the bulk and desorbing in a second-order process may complete the physical picture. It is well established that the rate of recombination depends strongly on the atomic structure of the surface and is e.g. generally higher on stepped surfaces than on flat close packed planes. In order to gain atomic level information on the real morphology of a surface after common bake-out and vacuum firing SS samples were imaged in the atomic force microscope (AFM) and the scanning tunnelling microscope (STM).

The main experimental work has been carried out on a combined STM – atom probe field ion microscope (AP-FIM) apparatus. The STM (Omicron STM-1) is directly attached to the UHV chamber which houses the field ion microscope with time-of flight mass spectrometer and position sensitive detection of the field evaporated ions (3D atom probe) [6]. A unique feature of the particular combined instrument is that it allows a fully-predictive preparation of STM probe tips in situ by FIM which is important for a reliable imaging of complex surfaces.

AFM and STM show that the high temperature treatment of SS in vacuum lead to a significant change in surface morphology which clearly can be seen in Figure 1 and 2. The surface after vacuum firing shows the formation of large flat terraces which can be assigned to (111) planes. These terraces are bounded by bunched steps and facets corresponding in orientation almost to (110) planes and (100) planes. The deep grooved grain boundaries (Figure 1) and facets formed by bunched atomic steps (Figure 2) represent very active sites for adsorption and recombination. A close up view on the large (111) terraces by STM show that there are a lot of vacancies too [7,8].

From STM and AFM data it can be concluded that the outgassing behaviour is almost controlled by surface defects and subsurface sites where hydrogen is deeply trapped. From this results a more complete description of the outgassing process may be given by a more or less dynamic equilibrium between diffusion, sojourn in different level traps and recombinative desorption.

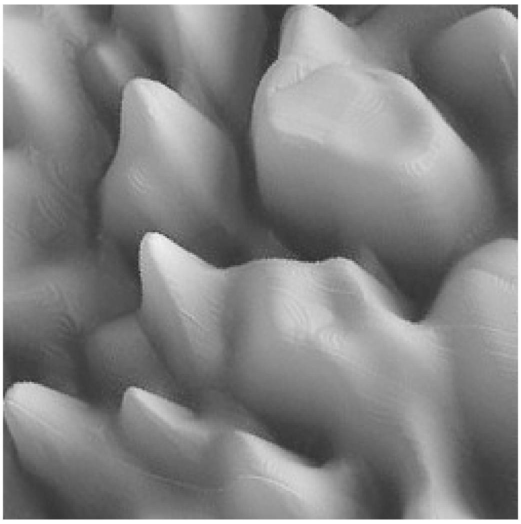

Figure 1: AFM micrograph (3D) of the SS surface after 20min@1250K vacuum firing. Image size 10 µm x 10 µm, z-scale enhanced.

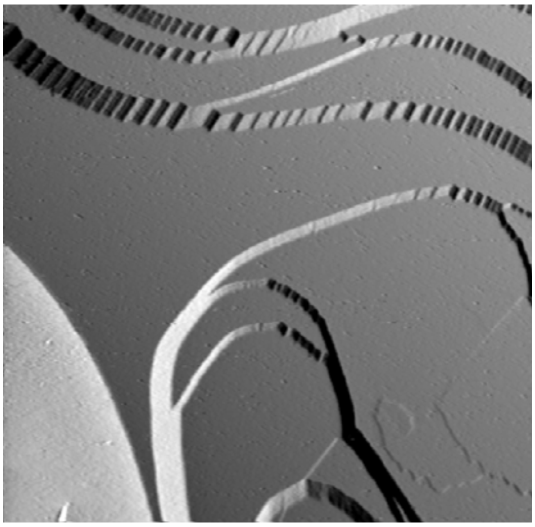

Figure 2: STM micrograph of the SS surface (top terrace) after 20min@1250K vacuum firing. Image size 1µm x 1µm (derivative).

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