INCONEL SURFACE COMPOSITION CHANGES DUE TO THE IMPINGEMENT OF ATOMIC HYDROGEN AT DIFFERENT TEMPERATURES

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ABSTRACT

The surface composition of high nickel alloy samples has been measured with Auger electron spectroscopy after heating the samples to temperatures from 150°C to 410°C and after their exposure to atomic hydrogen at these temperatures. The most remarkable effect of the hydrogen exposure is the removal of C at lower temperatures, accompanied by the segregation of S.

1. INTRODUCTION

The impingement of atomic hydrogen species onto the first wall of a tokamak is an important process of plasma wall interaction, among others because it may cause chemical reactions with the first wall material and its impurities. Especially, in tokamak cleaning discharges and in glow discharges, used for tokamak cleaning, the cleaning efficiency has been attributed to chemical reactions of atomic hydrogen species with those impurities of the first wall which form stable gaseous compounds with hydrogen, e.g. C_nH_m, H_2O (1). This is why we investigated surface composition changes due to the exposure to thermal atomic hydrogen. The material under test is a high nickel alloy, Nicrofer 7216 equivalent to Inconel 600, being the first wall material of TFR 600 (2), JET (3) and possibly of TEXTOR (4).

After identifying the removal of C and O due to the exposure to atomic hydrogen (5) we investigated the influence of the sample temperature on the surface composition changes by atomic hydrogen.

2. EXPERIMENTAL PROCEDURE

The apparatus has been described elsewhere (5). Briefly, in

a preparation system atomic hydrogen is generated by thermal dissociation of molecular hydrogen on a hot tungsten ribbon. After exposing the samples to certain atomic hydrogen doses, they are transferred by vacuum locks to an analysis system equipped with an Auger electron spectrometer. The samples are heated by the radiation of the ribbon and, in a controlled manner, by RF. Due to the radiation the lowest temperature adjustable is 150°C.

3. RESULTS

The samples are polished (8 µm) and cleaned with acetone and methanol just before their introduction into the vacuum system. Then blank runs are performed by heating the samples for half an hour at the desired temperature in vacuo and thereafter in molecular hydrogen at $5 \cdot 10^{-3}$ mbar. After every step the surface is analysed in order to separate the influence of the temperature treatment and of the molecular hydrogen exposure from that of the atomic hydrogen.

The initial surface composition in atomic % is as follows: Ni: 43...58, Cr: 2...3.5, Fe: 3...4, C: 21...36, O: 5...11, N: 1...2, S: 0.5...1, Cl: 1...2.5, Ca: 0.5...1.

For comparison, the bulk composition in atomic % is (6) Ni: $\geq$70, Cr: 16...19, Fe: 6...10, Mn: $\leq$ 1, Cu: $\leq$ 0.5, Ti: $\leq$0.4, Si: $\leq$ 1, C: $\leq$ 0.4, S: $\leq$ 0.03, P: $\leq$ 0.06.

The heat treatment changes the surface composition at low temperatures (150, 200°C) only by some percent, but at higher temperatures (260...410°C) significantly. Especially, Cr accumulates on the surface and its concentration after the heat treatment increases with the temperature. At 410°C it is even higher (23 at%) than the bulk concentration. Segregation of S is detectable at temperatures of 200°C and above, but is pronounced only at about 400°C. The concentration of O decreases during the heat treatment by some at % which is presumably due to the outgassing of water. The C concentration does not change significantly at temperatures up to 200°C, between 260°C and 360°C it decreases to 20...28 at% which is about 3/4th of its initial concentration and at 410°C it drops down to 6 at%. The concentrations of Ni and Fe remain essentially unchanged by the heat treatment.

A surface composition change due to molecular hydrogen has not been identified.

The most remarkable surface composition changes due to the exposure to atomic hydrogen are the removal of C and the segregation of S.

The removal of carbon by atomic hydrogen can be observed at

sample temperatures between 150°C and 260°C. Generally, there is a fast decay of graphitic C at low doses and a slow decay of carbidic C at higher doses. Graphitic and carbidic C are distinguished by its Auger peak shapes. The transitions from the fast to the slow decay anf from the graphitic to the carbidic C peak shape occur at the same hydrogen dose, which decreases with increasing sample temperature. The final C concentration is 2...5 at%. At higher temperatures (310...410°C) there is no significant decrease of the C concentration due to atomic hydrogen exposure. However, the heat treatment at 410°C reduces the C concentration to about 6 at%.

Sulphur segregates at the surface up to a concentration of 11 ...13at% at a dose of about 10^{19} cm^{-2}. At lower temperatures (150...260°C) the segregation is obviously connected with the exposure to hydrogen and the removal of C. At higher temperatures the segregation of S may be a temperature effect, mainly.

Some further information on the surface composition has been achieved by depth profiling of samples which have been exposed to a hydrogen dose of $10^{19} cm^{-2}$ at 150°C and to a dose of $5 \cdot 10^{19}$ cm^{-2} at 360°C. In both cases the Si is accumulated in a thin sheath which is completely sputtered with an argon ion dose of 10^{16} cm^{-2} and $2 \cdot 10^{15}$ cm^{-2}, respectively, the argon ion energy being 3 keV. Assuming a sputter yield S = 2 (7), these argon ion doses correspond to the sputter etching of 2,5 and 0,5 monolayers, respectively. In this depth the C concentration exhibits a maximum and decreases to the half of its maximum concentration after sputtering with doses of about $2 \cdot 10^{17}$ cm^{-2} and $2 \cdot 10^{16}$ cm^{-2}, corresponding to 50 and 5 monolayers, respectively. So the C is enriched in a much deeper sheath near the surface than S and the sheaths are much smaller at 360°C than at 150°C, which can be attributed to the faster diffusion at higher temperatures. Furthermore it turns out that the surface of the 150°C-sample is enriched with Ni and depleted with Cr, whereas the 360°C-sample shows the opposite behaviour.

Oxygen, finally, is at all temperatures reduced to about 1/3rd of its initial concentration by a hydrogen exposure of 10^{19} cm^{-2}. The corresponding final concentrations are 1...2,5 at%. The decay of the O concentration is rather low compared to that of the graphitic C. Therefore, we cannot conclude a chemical reaction of H and O from comparing the surface composition changes during the blank runs and the atomic hydrogen exposure. To do this other experimental arrangements which allow the detection of gaseous reaction product are needed.

4. CONCLUSIONS

If the question is to be answered what kind of and how many gas molecules leave the wall of a tokamak after a chemical reaction with atomic hydrogen, then the application of gas analysis methods is sufficient. If additionally a complete description of the surface composition is desired, it is necessary to take into account diffusion in the bulk material and segregation which requires the application of surface analysis and depth profiling techniques. Therefore, it is a rather difficult task to specify the surface which is really exposed to plasma wall interaction in a tokamak.

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