§16. Electrochemical Nitriding of Stainless Steel in Molten Fluoride Salt

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The structural material for liquid blanket system using molten fluoride salt such as FLiBe and FLiNaK needs corrosion resistance against the molten salts. According to thermodynamics, nitrides have well compatibility with molten fluoride salts. When nitride layer is formed at the surface of the structural material in the system, it is expected to avoid corrosion and keep soundness of the system.

Electrochemical nitriding for surface modification of structural material was tried in molten fluoride salt[1,2]. Experiment was carried out using specimen of stainless steel 316 (SS316) which is one of candidate structural materials. Figure 1 demonstrates the reactor for electrochemical nitriding. The specimen which was attached the end of the working electrode was immersed into the molten fluoride eutectic salt including Li_3N (molar ratio, Li_3N:LiF:KF = 2:49:49) at 600°C. In the nitriding treatment, the specimen was kept at +1.0V of a constant potential against the equilibrium potential of the AlLi alloy reference electrode in the molten salt by the potentiostat.

Figure 2 demonstrates nitrogen distribution in the cross section of the nitrided specimen by electron probe micro analyzer (EPMA). As shown in the nitrogen mapping image of Fig 2(a), after 240 minute treatment, nitrogen was introduced into a depth of 70μm from the surface. As shown in the line analysis for nitrogen in Fig.2(b), it was step-like distribution at the most deepest position where nitrogen was introduced. Figure 3 demonstrates X-ray diffraction (XRD) patterns. Those patters suggested that nitriding formed CrN and α-Fe_xN(x>8) and that, in the same time, initial austenite like structure (fcc structure) transformed to ferritic structure (bct structure). And in X-ray photoelectron spectroscopy (XPS) measurement, peak shift of Cr 2p3/2 binding energy also suggested formation of CrN as a main nitride product.

Finally, considering the experimental conditions such as temperature, nitrogen concentration and specimen composition, nitride formation was theoretically discussed based on combination of thermodynamics and electrochemistry. CrN, Cr_2N, Fe_2N and Fe_4N were considered from composition of SS316. Potential-nitride formation diagram and potential-nitrogen ion concentration diagram were made. From discussions on formation of these nitrides based on these conditions, it was theoretically derived that CrN is most stable. This theoretical consideration was well in agreement with the experimental result. In conclusion, these results demonstrate availability of this nitriding method and give a guideline for optimization of this nitriding process in molten fluoride salts.

1) Watanabe, T., Kondo, M. and Sagara, A. :Electrochimica Acta 58(2011)681.
2) Watanabe, T. :Ph.D. thesis submitted to Grad. Univ. Advanced Studies (2011).

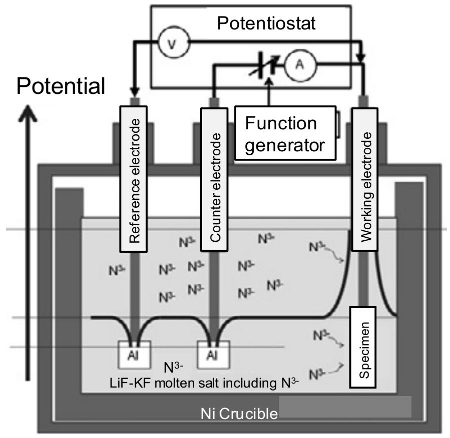

Figure 1. Reactor for electrochemical nitriding.

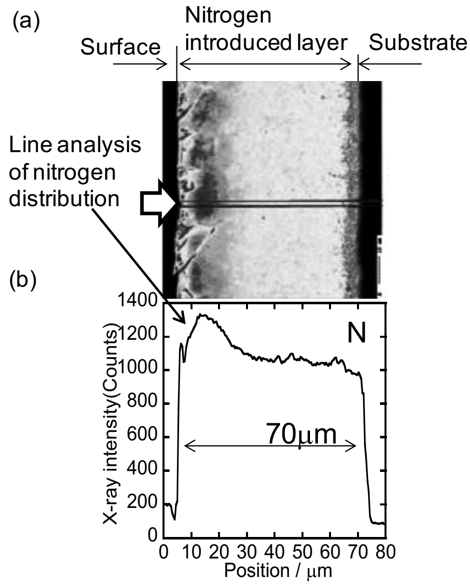

Figure 2. Nitrogen distribution observation of nitrided SS316 specimen by EPMA. Cross section of specimen treated for 240 min was observed. (a) Nitrogen mapping image, (b) line analysis of nitrogen distribution.

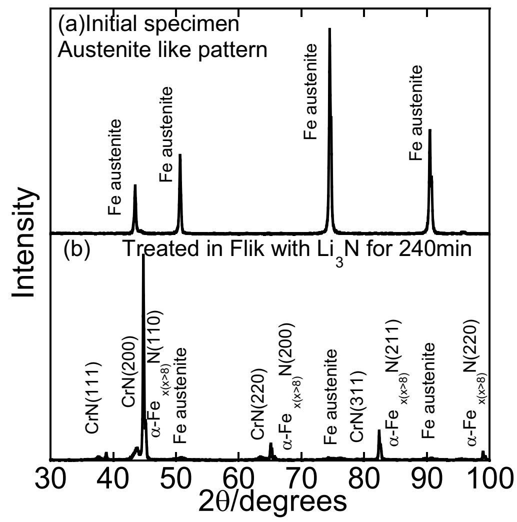

Figure 3. XRD patters. (a) Pattern of initial SS316, (b) pattern of nitrided SS316 specimen.