10. Task １０ Development of Cryogenic Materials Technology

10.1. R & G Goals

10.1.1 Goals of Phase II

The goals of phase II are to test material properties under liquid hydrogen environment and to develop elemental technology related optimized welding method. The material characteristic database will also be enhanced.

10.1.2 Goals for FY 2001

(1) Evaluation test will be implemented on the base metal and weld metal selected as candidate materials in the temperature area of liquid hydrogen, and basic material characteristic data will be enhanced. Evaluation on thin walled material will also be studied.

(2) Properties will be improved by conventional welding method, and elemental technologies will be developed with respect to new welding methods such as reduced pressure electron beam welding and friction stir welding.

(3) Additional data of the cryogenic material characteristic database will be input for enlargement of the database.

(4) The material structure, chemical compositions, transformation behavior and mechanism of low temperature embrittlement and hydrogen embrittlement will be clarified.

10.2 Results of FY 2001 Research and Development

In Phase II of the Research and Development, evaluation was made on the material characteristics of the base material and weld material in the low temperature including the liquid hydrogen environment using the selected candidate materials (austenitic stainless steel: SUS304L, SUS316L, SUS316LN, aluminum alloy: A5083A and A5454). As a result, it was found out that, while the selected base material maintained sufficient properties in liquid hydrogen environment, improvement of toughness was important for the weld metal. Accordingly, from the viewpoint of improving toughness, evaluation has been made on the applicability of new welding methods and welding materials that are emerging in recent years, and their effects have been verified.

In FY 2001, we have evaluated new welding methods and welding materials by expanding the scope of metal types to be applied. At the same time, we have analyzed the factors affecting toughness at a low temperature and clarified the mechanism of embrittlement. Conventional tests of material characteristics such as fatigue characteristics have been continued to enhance the database.

The following describes the major achievements obtained from the project of this fiscal year:

10.2.1 Evaluation of stainless steel material characteristics

It has been known so far that, stainless steel weld material, the presence of even a small quantity of δ-ferrite serves as a fracture propagation path at a low temperature, with the result that toughness is reduced. However, the δ-ferrite is essential to prevent high-temperature fracture in the welding operation, and the problem is how to ensure compatibility between the low-temperature toughness and prevention of welding fracture. By the end of the previous fiscal year, we demonstrated that SUS304 and SUS316L had a high degree of toughness at a low temperature despite the presence of δ-ferrite according to the reduced pressure electron beam welding method, and the TIG welding by high manganese based perfect γ- type welded metal characterized by reduced susceptibility to fracture at a high temperature had an excellent toughness at a high temperature and welding fracture preventive function.

In the program for this fiscal year, reduced pressure electron beam welding method has been applied to the high-strength SUS316LN effective in reducing the weight of a transportation container, and characteristics at a low temperature and hydrogen susceptibility have been evaluated. Further, perfect γ-type welding metal and TIG welding was also examined for hydrogen susceptibility using SUS316L.

Fig. 10.2.1-1 shows the Charpy absorbed energy and fracture toughness value (K_{IC}) of reduced pressure electron beam (RPEB) of SUS316LN. Although SUS316LN involves the problem of easier generation of blowhole-shaped welding defects caused by a high nitrogen content in the base material, the sound portion of SUS316LN ensures much higher toughness at a low temperature than that in the TIG welding method, similarly to the case of SUS304L and SUS316L. Further, neither reduction in toughness that would be caused by hydrogen charging nor remarkable susceptibility to hydrogen embrittlement were observed.

Fig. 10.2.1-2 shows the toughness of perfect γ-type weld metal and TIG weld metal (JJ1) of the SUS316L at a low temperature. It exhibits higher Charpy absorbed energy and K_{IC} than these in the conventional high δ-ferrite TIG welding method. Further, no reduction of toughness caused hydrogen charging was observed.

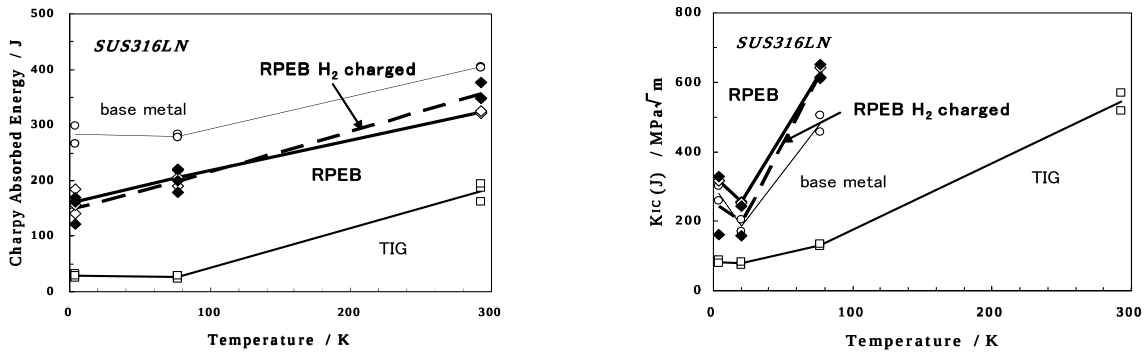

Fig. 10.2.1-1 Charpy impact properties and fracture toughness of SUS316LN reduced electron beam welding metal

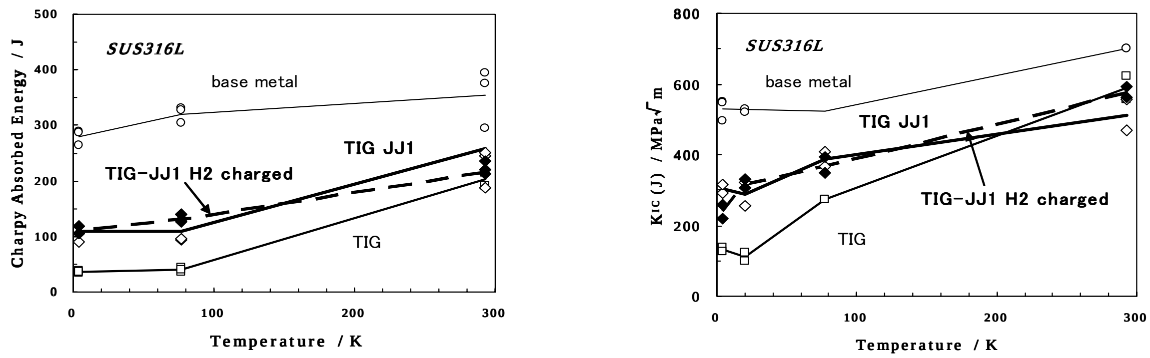

Fig. 10.2.1-2 Charpy impact properties and fracture toughness of SUS316L TIG welding metal

Following the project in the previous fiscal year, we have conducted a fatigue test of SUS304L, SUS316L and SUS316LN base metals and weld metals, and have enhanced the database. As a result, it has been found out that all base metals have a fatigue life of 10^6 to 10^7 times or more against fatigue under a higher stress equivalent to 0.2% proof stress. However, some of the TIG weld metals have a shorter fatigue life against fatigue, depending on the weld joint manufactured, and this defect has been estimated to be caused by very small fracture defects due to solidification.

10.2.2 Evaluation of aluminum alloy material characteristics

By the end of the previous fiscal year, we demonstrated that friction stir welding (FSW) improves the low temperature toughness of the aluminum alloy through the formation of a fine structure on the weld metal. It has also been made clear that the toughness can be improved by reducing the magnesium content of the base material. In the program for this fiscal year, friction stir welding (FSW) was applied to low-magnesium (3%) A5454 alloy, and the result were compared with that of A5083 with magnesium content of 5% obtained in the previous fiscal year and before. This is given in Fig. 10.2.2-1 In both the Charpy absorbed energy and K_{IC}, A5454 exhibits higher low-temperature toughness values than those of the A5083. It should be noted that, for the Charpy absorbed energy of A5454 according to FSW method, no fracture was caused by the impact of the test equipment used in the present experiment at any temperature. The actual value is estimated to be greater than this value.

In the meantime, whereas reduction of the magnesium content improves the low-temperature toughness, it decreases the tensile strength. To keep tack of the balance between them, we used a new A5086 alloy of 4% magnesium content to examine the characteristics of the large current MIG weld metal this year. The result is given in Fig. 10.2.2-2 It indicates that the strength and tensile strength are reduced, and K_{IC} value is increased with the decrease of the magnesium content. Observation of the fracture surface suggests that improvement of the toughness is attributable to reduction in the grain boundary segregation due to decreased magnesium content and improvement of the transgranular deformation. Further, the reduction of tensile strength in the A5083 can be explained by earlier fracture having occurred for the same reason due to high magnesium content.

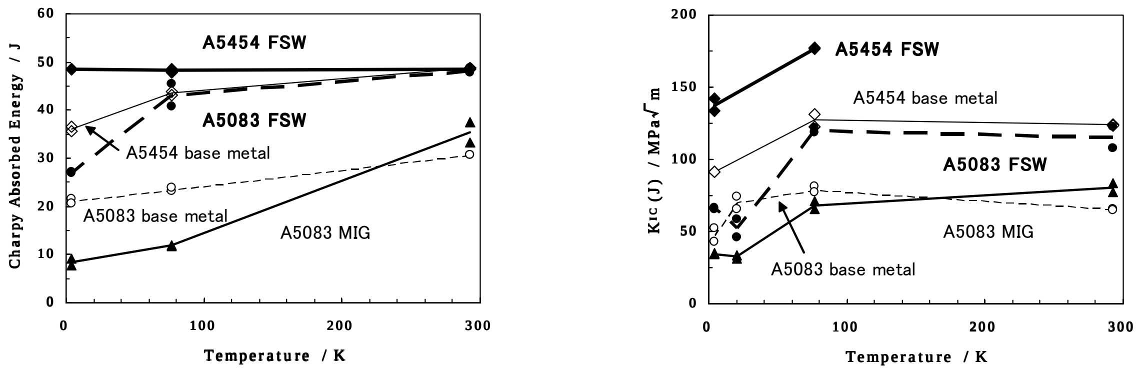

Fig. 10.2.2-1 Charpy impact properties and fracture toughness of A5454 friction stir welding metal

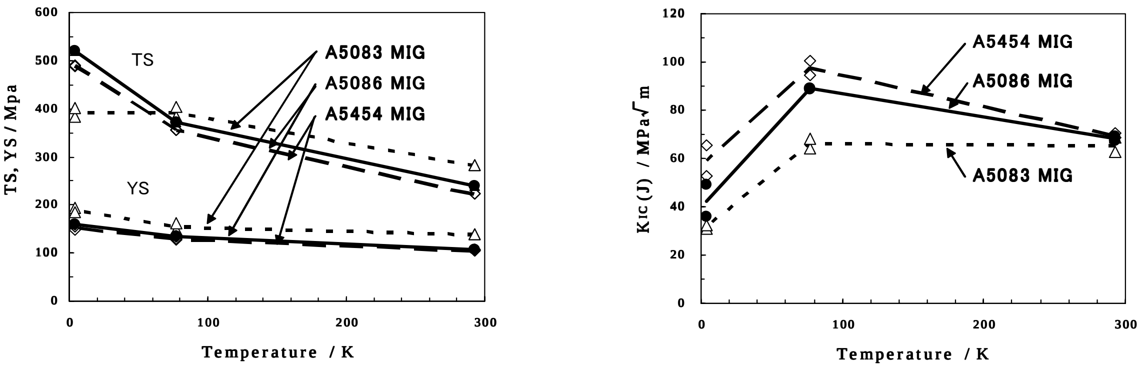

Fig. 10.2.2-2 Influence of magnesium content in aluminum alloy upon tensile strength and fracture toughness

10.2.3 Study of low temperature and hydrogen embrittlement in stainless steel weld metal

In TIG welding method, the thick-walled material is welded by multi-path welding process. Thermal impact in this case may affect the low-temperature toughness. Fig. 10.2.3-1 shows the result of examining the Charpy absorbed energy by heat treatment of the PREB material based on high-energy 1-path welding method. As the temperature is higher and keeping time is longer under the heat treatment conditions, the TIG weld metal and Charpy absorbed energy are reduced; thus, the reduction in toughness has been verified. In the subsequent program, we are planning to observe the fracture surface and examine the structure, thereby clarifying the causes of these phenomena. It should be added that, in the program for this fiscal year, we also conducted a precision fracture toughness test to clarify the difference in fracture toughness depending on the position of the weld metal, and checked the low-temperature embrittlement behavior.

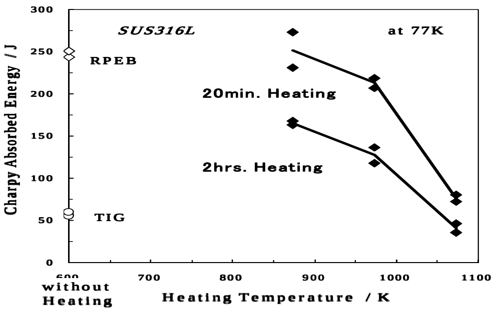

Fig. 10.2.3-1 Influence of thermal treatment upon Charpy absorbed energy of stainless steel

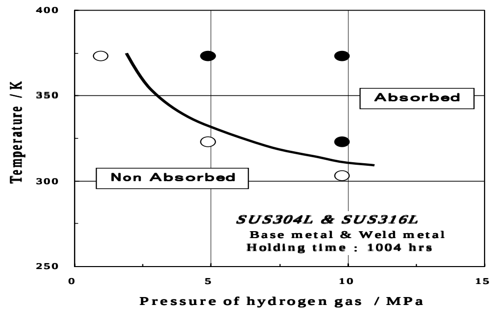

Fig. 10.2.3-2 Conditions on the intrusion of hydrogen into stainless steel

Upon intrusion of hydrogen into stainless steel, characteristics may be deteriorated in some cases. Fig. 10.2.3-2 shows the result of exposing the stainless steel for a long time in a hydrogen gas atmosphere at various temperatures and pressures to examine if hydrogen enters or not. The critical pressure for hydrogen intrusion at 100 ℃ is estimated at 1 to 5 MPa. The higher the pressure, the smaller the critical temperature for hydrogen intrusion. Our further task is to pay attention to the storage of over-20MPa high-pressure hydrogen gas and to examine the possibility of hydrogen intrusion and its influence upon the material. It should be noted that the relationship between the embrittlement in the hydrogen gas environment and stainless steel components has also been studied, following the study project for the previous fiscal year.

10.3. Summary and future development issues

We have evaluated new welding methods effective in improving toughness at a low temperature by expanding the scope of the metal types to be applied. Reduced pressure electron beam welding of stainless steel has been shown to provide the same high toughness at a low temperature in high-strength SUS316NL as in other types of steel. No influence of hydrogen charging was observed. Perfect γ-weld metal TIG welding method has also shown to provide a high degree of toughness. No influence upon hydrogen susceptibility was observed. Further, application of the friction stir welding to the A5454 aluminum alloy of low-magnesium content has been found to ensure a drastic improvement in the low-temperature toughness. In this project, we could also extend our knowledge on the strength/toughness balance by magnesium content.

Since development of dispersed use at the hydrogen supply station and others is expected to be accelerated and promoted, our subsequent task will be to evaluate the characteristics of the thin-walled material for small- to medium-sized containers and to evaluate and study the embrittlement characteristics under high-pressure hydrogen gas environment.