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Microstructure and Mechanical Properties of Friction Stir Welded High-strength Steels

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論文内容要旨

FSW has been widely studied and commercially applied with a focus on low softening temperature material (LSTM). T^h~ feasibility of FSW of high softening temperature material (HSTM) has been also reported relatively early. Some initial feasibility studies FSW of 12% Cr alloy and low carbon steel are demonstrated by TWI. Thereafter FSW feasibility was examined in several types of HST such as ferritic steels, stainless steels and heat resistant steels. Transverse tensile specimens failed in regions corresponding to the base mate and their transverse tensile properties are governed by the base material properties in most of FS-welded steels, *i.e.*, yield and ultimate tens strengths of the weld were comparable to those of the base material. However, there was a limitation of applying FSW to steels because lack of proper tool. Recently, new tooling made of polycrystalline cubic boron nitride (PCBN) that appears capable of meeting th requirements, especially, the wear resistance, was developed.

Sato *et al.* have showed that FSW can be applied to ultrahigh carbon steel successfully. And Tracy W. Nelson *et al.* and A. Kumar *et* have researched about friction stir welding for API steel. Previous research of friction stir welded steels indicated possibility of friction stir welding of steels and most of the SZ of friction stir welded carbon steels have brittle phase formation at the weldment. And, recently there is some report friction stir welding for HSLA steel. However, microstructure and weld properties of FSW high strength steels with high carbon have not been fully definitized. Additionally research for the advanced weld properties is needed.

In the present study, FSW was applied to API K55 and P110 grade steels for casing coupling to apply to drilling using PCBN tool and Q60 tool. The microstructures were observed using electron microscopy. Their microstructural evolution and its effect on several properties were discussed in FS welds of these high strength steel with high carbon.

The microstructure and mechanical of FS welded API K55 which is high strength steel with high carbon contents were evaluated. To setup the proper welding parameter, trial welding was conducted. With those results of trial welding, FSW of API K55 was conducted. Sound welds of API K55 could be produced by FSW regardless of welding parameters. The SZ exhibit a significant increase of hardness regardless of rotational speed of tool. The microstructure of SZs consisted of martensitic phase predominantly. Ferrite and pearlite of base material transformed to single austenite phase, and this austenite undergo solid state transformation to martensite, because of the high hardenability (C eq=0.631). This result also exhibits that the peak temperature exceeds A_3 temperature at the SZs of welds.

Tensile properties of welds matched its base material's yield strength and ultimate tensile strength and there was almost no effect on the tensile properties according to changing heat input by changing rotational speed of tool. Toughness of welds was much lower than that of base material at all temperature regardless of rotational speed of tool. These results would be explained with microstructural distribution that hardened phase, martensitic phase composed welds predominantly.

There was the microstructural inhomogeneous region which consisted of more bainitic ferrite at the AS in the SZs than other region in the SZ. The formation of this region was affected by higher peak temperature and slow cooling rate at the AS respectively.

API P110 grade steel which is high strength steel with high carbon content consisted of tempered martensite as received condition. The antecedent trial welding was conducted to set the proper welding parameter for FSW of API P110. In the welds, the hardness gradually decreases from the base material region toward the boundary between the HAZ and the SZ, and increases significantly in most part of the SZ. The used welding parameters have no influence on the hardness in the SZ. There was further tempering during at the HAZ during FSW process. During further tempering, tempered martensitic phase transformed to ferrite and globular cementite. The peak temperature of the softest region of the HAZ would be just below A_1 temperature. This further tempering caused the decrease of hardness at the HAZ. The microstructure of SZs of the welds consisted of most of martensite and little bainitic ferrite. This microstructure suggests that peak temperature of the SZs exceed A_3 temperature during FSW process.

The UTS, YS and El of the welds are lower than those of the base material. All fracture occurred at the HAZ regardless of rotational speed. The softest region which has the lowest elongation acts as deformation concentration region. In case of fracture toughness, the HAZ has the highest toughness property in the welds, including base material.

Microstructural inhomogeneous region was also observed in the SZ regardless rotational speed. The principal possibility for a formation of this region could be presumed as boron segregation and thermal history. The results indicated that the key factor for formation of microstructural inhomogeneous region is not contribution of boron segregation but thermal history.

Previous research showed that the hardened phase (Martensite + Bainitic ferrite) formed in the SZ during FSW process, and it led to a deterioration of mechanical properties of welds. To suppress the formation of hardened phase, the rotational speed of tool with Q60 tool was decreased for decreasing heat input. SZs of both steels exhibit decrease of hardness as decreasing heat input, which revealed a decrease of martensite. Particularly, there was no martensite formation in the SZ of API K55 weld without any defect produced by 50rpm rotational speed of tool. There was also the increase of hardness in the SZ, and it would be result of grain refinement by recrystallization. Fracture toughness of 50rpm specimen was higher than that of SZ, even higher than that of base material because of no martensitic transformation and grain refinement due to recrystallization.

In welds of API P110, it was possible to decrease the martensite formation during FSW process without any defect. There was no drastic increase of hardness at the SZ, although decrease of hardness at the HAZ also accrued because of further tempering during FSW.

Microstructure of the SZ consisted of martensite, ferrite and bainitic ferrite. The fraction of martensite was around 10% which is much smaller than that of SZs produced by PCBN tool. This result suggests that the peak temperature of SZs was between A_1 and A_3 temperature during FSW.

There is no effect of rotational speed of tool which is related to heat input change on tensile properties and all fracture occurred at the HAZ during tensile test. It was result from the lowest yield strength at the HAZ in the weld. Fracture toughness of the HAZ the highest value in the welds, even higher than that of the base material.

In this study, the feature of martensite produced FSW was evaluated. Because FSW is a hot deformation process, most of the deformation occurs at elevated temperatures. Therefore, martensitic transformation during FSW process would be affected by not only thermal, but also strain effect. In case of prior austenite grain size, there is an inclination of decreasing the prior austenite grain size as decreasing the rotational speed of tool and it would be result of suppression of dynamic grain growth by decreasing heat input. Martensite block size also evaluated. The results indicated that there is an inverse proportion relation between rotational speed and block size, and proportion relation between prior austenite grain size and block size. Misorientation angle distribution of grain boundary of SZ exhibit that there is inclination to increase the Group 2 variant pair which has 49.5 ° misorientation angle. The result would be affected by residual stress.

The feasibility of FSW was confirmed in API K55 and P110 which are high strength steel with high carbon content. It is difficult to apply conventional fusion welding to those materials for drilling, because of high carbon equivalent. The sound welds could be produced successfully by FSW.

In the welds of API K55 produced by FSW, duplex microstructure of ferrite and pearlite transformed to martensite and bainitic ferrite in the SZ. The massive martensite transformation is originated from austenite experiencing dynamic recrystallization at elevated temperature, subsequent rapid cooling and large strain induced by frictional stirring. The large strain affected on austenite grain size at elevated temperature and austenite grain size became smaller than that of fusion welding. This refining austenite grain size led to decrease of block size, because the austenite grain boundary predominate the nucleation site of martensite. There would be effect on enhancing mechanical properties of martensite. Although martensite block size refined which would be effect on mechanical properties, hardened phase make mechanical properties deteriorate. The lower rotational speed of tool suppressed the formation of martensite in the SZ and there was no martensite phase in the SZ produced by 50rpm. The pole figure of SZ indicated bcc shear texture. It showed that the microstructure formation affected by shear stress occurred by tool rotation during process. These refined ferrite and globular cementite enhanced tensile properties and fracture toughness.

In API P110 high strength steel, FSW produced sound welds successfully at all welding parameters used in this study. The high rotational speed caused very high hardness in the SZ due to formation of high fraction of quenched martensite, but the decrease in rotational speed could reduce hardness and fraction of quenched martensite in the SZ. This result implies that high toughness and low hydrogen-induced cracking susceptibility could be achieved in the SZ at the low rotational speeds. However, all welds underwent more tempering of the base-material microstructure at the HAZ, which acted as the fracture site of the weld during the transverse tensile test. Since the fracture locations exhibited roughly the same hardness and microstructure, the transverse tensile properties of the weld hardly depended on the welding parameters.

This paper clarified the microstructural features and the microstructural evolution in FS-welded high strength steels with high carbon contents and showed the relationship between microstructure and mechanical properties. FSW is solid state joining technique without melting of base material and will provide benefits in the chemical, oil, gas and power plant industries having problems in weld joints of carbon steel. The desirable weld joint will reduce the risk often caused in large structures containing many weld joints.

Since this is an initial study systematically performed on FSW of high strength steel with high carbon content, there are many unclear issues concerning with microstructural phenomena and other corrosion properties and mechanical properties. One of the major problems is formation hardened phase in the weld which deteriorates the mechanical properties. To suppress the hardened phase, it is need to conduct FSW with low rotational speed. However it makes tool wear or fracture during FSW process. Of course it is possible to solve the hardened phase formation and deterioration of mechanical properties by post-weld heat treatment (PWHT). However it is difficult or impossible to apply the PWHT at drilling field where these API K55 and P110 were used at. The hybrid welding of precedence of conventional fusion welding and microstructural modification by FSW can be considered to solve those problems. The present study evaluates the martensite morphology. There was an evidence of different martensite variant selection of the martensite produced by FSW compared to that of martensite produced by conventional fusion welding. It would affect mechanical properties of welds.

As mentioned above, there are many other issues to be solved and improved in FSW of steels, in order to use friction stir welding commercially on welding of high strength steel with high carbon e structures in the field, especially drilling field. It is clear that advanced research about FSW of API steel for drilling will propose extraordinary welding procedure to join pipe for oil and natural gas drilling and transportation. The effective application of FSW will provide the new progress in the petroleum industry.

論文審査結果の要旨

石油掘削用に開発された高強度鋼は溶融溶接性に劣るため、一般に溶接施工は行われない。溶融溶接時の問題は、高入熱もしくは溶融・凝固に起因することから、低入熱の固相接合法である摩擦攪拌接合の適用によりその防止・低減が期待される。本研究では、API 規格の高強度鋼である K55 鋼と P110 鋼に対して摩擦攪拌接合を実施し、接合部のミクロ組織と機械的特性の関係、接合部組織と機械的特性に及ぼすツール回転速度の影響ならびに形成組織の材料組織学的特徴について明らかにすることを目的としている。論文は全編６章で構成されている。

第１章は序論であり、本研究の背景および目的を述べている。

第２章では、K55 鋼摩擦攪拌接合部におけるミクロ組織と機械的特性の関係について調べている。摩擦攪拌接合により攪拌部には焼入れマルテンサイトが生成するため著しい硬さ上昇が生じ、継手強度は母材と同等であるが、攪拌部において靭性低下が生じることを明らかにしている。

第３章では、P110 鋼摩擦攪拌接合部におけるミクロ組織と機械的特性の関係について調べている。攪拌部にはマルテンサイトが生成する一方、熱影響部ではマルテンサイトの更なる焼戻しが生じるため、攪拌部での硬さ上昇と熱影響部での硬さ低下が生じることを示している。継手強度は母材よりも低下し、攪拌部における靭性低下が生じることを明らかにしている。

第４章では、攪拌部の靭性向上を目的として、低速度のツール回転を用いた摩擦攪拌接合を実施し、その接合部のミクロ組織と機械的特性について調べている。ツール回転速度の低下により、攪拌部におけるマルテンサイト形成を抑制でき、母材以上の靭性を付与できることを明らかにしている。

第５章では、摩擦攪拌接合により得られたマルテンサイトの材料組織学的特徴について調べている。通常の溶融溶接部に比べてブロックサイズの小さなマルテンサイトが得られ、結晶学的に特徴的なバリアントの存在を明らかにしている。

第６章は本研究の結果をまとめた総括である。

以上要するに本論文は、高強度鋼摩擦攪拌接合部の組織と機械的特性ならびにそれらの関連性を明らかにして、良好な機械的特性を得るための組織制御と接合条件の指針を提示したものであり、材料システム工学の発展に寄与するところが少なくない。

よって、本論文を博士（工学）の学位論文として合格と認める。