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Korrosion unter Wärmeübergang der rostfreien CrNi-Stähle DIN W. Nr. 1.4306n und 1.4361 ESU in azeotroper Salpetersäure

<u>Kurzfassung</u>

Die Salpetersäure-Korrosion der austenitischen rostfreien CrNi-Stähle DIN W. Nr. 1.4306 n (AISI Type 304 L) und 1.4361 ESU wurde unter Wärmeübergang bei 120 - 150 °C geprüft. Dieser Temperaturbereich entspricht in etwa dem einer inneren Auflöseroberfläche, die von außen dampfbeheizt ist, und als Teil der wichtigsten Komponente des Purex-Prozesses dazu dient, abgebrannten Kernbrennstoff in mittelkonzentrierter Salpetersäure aufzulösen.

Für diesen Zweck wurden zwei Laborprüfstände gebaut, um 5 mm-Blechproben, die somit einerseits elektrisch beheizt, andererseits Salpetersäure-exponiert waren, zu testen. Auf diese Weise konnte ein Wärmefluß in einem Temperaturgradienten von 10 - 40°C zwischen der Metalloberfläche (120 - 150 °C) und der Säure (110 °C) eingestellt und langzeitig aufrechterhalten werden.

Die Ergebnisse beider Stähle zeigten, daß das Korrosionsverhalten der Metalloberflächen unter Wärmeübergang bei Temperaturen am oder oberhalb des Siedepunkts der azeotropen Säure (120 °C) grundsätzlich durch ähnliche oder gleiche Beziehungen vom Arrhenius-Typ beschrieben werden können, wie sie bei isothermen Immersionsexperimenten im Temperaturbereich 0 - 120 °C gemessen und in der Literatur wiedergegeben wurden. Die Oberflächentemperatur des Metalls war der Korrosionsgeschwindigkeits-bestimmende Parameter.

Hinweis:

Dieser Vortrag wurde auf dem Internationalen Symposium "Korrosionsbeständige Legierungen 1991" gehalten, das vom 1. - 5. September 1991 am Institut für Gießereiwesen der Hochschule für Bergbau und Metallurgie, Krakau/Polen durchgeführt wurde.

Corrosion under Heat Transfer of the CrNi Stainless Steels DIN W.Nr. 1.4306 n and 1.4361 ESU in Aceotropic Nitric Acid

<u>Abstract</u>

The nitric acid corrosion of the austenitic CrNi stainless steels DIN W.Nr. 1.4306 n (AISI Type 304 L) and 1.4361 ESU was tested under heat transfer at 120-150 °C. This temperature range corresponds about to that of the nitric acid exposed inner surface of a dissolver which is externally heated by steam to dissolve as part of the Purex-process - spent nuclear fuel in nitric acid of medium concentration.

For that purpose two laboratory apparatus were constructed to test 5 mm thick sheat specimens while they were electrically heated on one side and at the other side exposed to aceotropic nitric acid. Thus, a heat flow in a temperature gradient of 10 to 40 °C between the metal surface (120 - 150 °C) and the acid (110 °C) could be established and kept constant on long-term.

The results of both steels showed that the corrosion behavior under heat transfer at metal surface temperatures at and above the boiling point of the aceotropic acid (120 °C) can principally be described by similar or equal Arrhenius type relationships as measured by isothermal immersion experiments within the temperature range 0 - 120 °C and given in the literature. The surface temperature of the metal was the corrosion rate determining parameter.

Notice:

This paper was presented to the International Symposium "Corrosion Resistant Alloys 1991", organized by the Institute of Foundry Engineering, Academy of Mining and Metallurgy, Cracow/Poland, September 2-5, 1991.

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1. INTRODUCTION

Differences in temperature are the prerequisite for heat flow. Heat flows through metallic walls of vessels, containers, pipes etc. if temperature gradients are maintained between both, the inner and outer metal surfaces. Besides thermomechanical stresses in the wall, differences in surface reactivity have to be considered in respect to metal corrosion caused by aggressive media and/or heating/cooling fluids.

1 -

The risk of accelerated corrosion at heat transfer surfaces [1] can be caused by

- non-uniform heating or heat removal, hot spots, overheating etc.,
- scale or deposit formation by corrosion products, non-soluble constituents or decomposition of fluids,
- flow velocity of the heating or cooling fluid,
- effects of localized corrosion at seals (crevices) or at different metal contacts,
- phase boundary effects at gas/liquid interfaces.

As part of an extensive corrosion study by application of chemical and electrochemical testing methods (Fig. 1) the present work contributes to an appropriate selection of construction materials for reprocessing of spent nuclear fuel by corrosion testing of various austenitic CrNi steels corrosion under simulated operation conditions of a steam-heated dissolver unit (Fig. 2).

During service the outer side of the dissolver is heated by steam to 130 - 160 °C, heat flows through the dissolver wall and brings the process medium up to temperatures which allow the dissolution of the fuel/fission product in half-concentrated nitric acid, mainly under boiling conditions (110 - 115 °C).

In our experimental approach the conditions of material exposure were simulated by a flat steel specimen pressed at one side against a heated steel bloc, at the other side being exposed to hot nitric acid, stirred for better acid diffusion to the specimen and better heat removal from the acid (temperature control).

In Fig. 3 the temperature distribution in the wall of a dissolver is compared with its experimental laboratory simulation.

2. EXPERIMENTAL

2.1 Test Devices

As shown in Fig. 4 a testing device was designed in which metallic specimens could be tested under heat transfer in four positions of specimen exposure. Fig. 5 shows the selected version for the initial tests. The heat was generated by four resistance heated cartridges (4 x 750 W) positioned within a heating bloc. It was transfered to the square metal specimen ($72 \times 72 \times 5 \text{ mm}$) pressed on it. A paste for improvement of the thermal contact was used. The test specimen on its other side was connected to a 0.7 ℓ acid containing glass flask, a seal was positioned between both and a clamp kept the whole arrangement tightly together. Heat in-put into the metal bloc was controlled by a thermocouple, heat transfer to the acid exposed specimen surface was checked by two other ones positioned in narrow boreholes about 0.5 - 1 mm below that surface.

The glass flask contained a spiral cooler and a thermocouple for thermostatically controlled heat removal from the acid. Other glass tubes connected the flask to a stirrer, to an acid vapor condenser, and - mainly for the tests of W.Nr. 1.4361 ESU - to an acid redistillation/purification (reflux) system (Fig. 6).

2.2 Plastic Seals

Under long-term testing a sufficient resistance of the seal against nitric acid attack was required when it was positioned between the test specimen and the acid containing flask. Preliminary tests during 100 - 165 h in boiling aceotropic nitric acid (Tab. 1) showed that 4 plastic ring shaped seals - from which finally Teflon was used - fulfilled the above mentioned requirements. When used for testing of CrNi steel W.Nr. 1.4306 n specimens no sealing material was able to avoid crevice corrosion by nitric acid.

2.3 Test Materials

The chemical composition of four steels is given in Tab. 2.

Mainly two CrNi stainless steels were tested under heat transfer:

- W.Nr. 1.4306 n / batch No. 681870
- W.Nr. 1.4361 ESU / batch No. G 57128

The other two steels were tested for reasons of comparison only.

Test specimens of $72 \ge 72 \ge 5$ mm in size were fabricated from steel plates. The exposure to the acid was performed on 70 % of one side which equals about 40 cm². The specimens were polished mechanically by emery paper, then electropolished to remove a layer of about 20 µm metal from the surface. In a cleaned and dried condition the specimens were weighed before and after testing.

2.4 Test Acid

According to the standards of intergranular corrosion testing of CrNi steels in oxidizing acids [2] the aceotropic 14.4 m nitric acid was used at 120 °C as test medium.

2.5 Test Procedure

After all above mentioned parts were put in position, the apparatus was heated without acid for 1 h to 120 °C to get the seal by creep suitably tight; if necessary the clamp had to be re-tightened. Thus a slow in-put of cold acid avoided thermoshocks to the stressed system. An additional heating tape helped to bring the acid quickly on test temperature (110 °C). Then time was taken to measure the duration of the exposure. Testing was performed under heat in-put and out-put, redistillation and condensation of the aceotropic acid.

2.6 Test Evaluation

By weighing before and after the test, occasionally during test interruption, the metal loss by acid corrosion was measured and calculated in terms of mg dm⁻² d⁻¹. Metallographic cross-sections and surfaces of the corroded specimens as such were evaluated by light microscopy.

3. TEST RESULTS

3.1 W.Nr. 1.4306 n (18Cr 11Ni LC)

This material is the standard construction material in Purex process engineering and therefore a reference material for various of our chemical and electrochemical tests [3]. Its corrosion behavior under the applied isothermal standard test conditions (Huey-Test) - as given in Fig. 7 - is from the engineering point of view not satisfactory since a continuous increase of the corrosion rate was measured through the total duration of testing (15 periods x 48 h = 720 h) and came within the last period to 0.7 mm/a. The task was to check whether a noticeable increase of the corrosion rate would occur under heat transfer: first of all by setting the surface temperature to 120 °C, a temperature at which isothermal and heat transfer corrosion rate could be directly compared, then by increasing it stepwise up to 150 °C.

The results (Fig. 8) of the CrNi steel W.Nr. 1.4306 n at 120 °C showed during the first three test periods which equals an exposure of 144 h a reasonably good agreement of both test results, under isothermal and heat transfer conditions. After this time the reaction rate experienced a drastical increase. Its acceleration by increase of temperature (a matter of being tested here) was overrun by a superimposed, strong and local metal loss which developed in the crevice between the specimen and the ring seal, forming - besides a uniformly corroded central surface area - a deep ditch below the seal. Therefore the gravimetric evaluation of all tests between 120 and 150 °C had to be focussed on timely reduced exposures of 3 x 48 = 144 h duration. The results are given in Fig. 9 and 10 as function of time and temperature. In Fig. 11 our results are compared with those of E.-M. Horn and K. Schoeller [4] who tested isothermally the electroslag remelted version of W.Nr. 1.4306 s ESU. Adding our results (120 - 150 °C for 3 periods of testing) to the Arrhenius diagram of the above mentioned authors, it becomes obvious that the temperature dependence (expressed as slope of the Arrhenius function) is about equal, but that in case of our results a quality - related shift to constant higher metal losses was found.

Fig. 12 - 15 shows LM-micrographs of that steel after nitric acid corrosion (144 h, 150 °C) in cross-section and from the surface as such. The normal type of intergranular (preferential grain boundary) attack and of grain losses from the surface become visible. The heavy superficial attack leaves only some etched grains in the original surface on which the microscope was focussed.

3.2 W.Nr. 1.4361 ESU (17Cr 14Ni 4Si)

The aceotropic (14.4 m = 65 %) nitric acid has - in respect to the corrosion rate of this 4 % Si-containing CrNi steel - the highest aggressivity (Fig. 16). Our isothermal Huey-test results (Fig. 17) confirmed those published by E.-M. Horn and K. Schoeller [4] (Fig. 18). All of results agree especially in respect to a relatively high, but constant reaction rate as function of time. Expecting that crevice corrosion is no problem when this material is tested under heat transfer (the corroding material builds up a protective SiO₂ surface layer [5,6]) 720 h test were performed. The test conditions became more constant and precise by application of the redistillation/purification method (Fig. 6) excluding corrosion products (to avoid interference by rising redox potential) but also by not interrupting the tests (no acid renewal required). The test results - as given in Fig. 19 and 20 as function of surface temperatureshowed an exponential increase of the corrosion rate, a fact which fits perfectly - as an extension to higher temperatures - to the above mentioned Arrhenius plot of Horn and Schoeller's isothermal results of W.Nr. 1.4361 ESU for temperatures between 0 -120 °C (Fig. 18) [4] and the given equation for the logarithm of the linear reaction rate W as function of absolute temperature T:

$$\ell g W_{\ell in} = 8.2 - \frac{3290}{T} [mm/a]; E_A = 63.0 \, kJ/mol$$

In Fig. 21-24 LM-micrographs of the corroded surface in cross-section and from the surface as such show the very rough, but homogeneously distributed thin SiO_2 surface layer, but no sign of intergranular corrosion. The corrosion attack took place in an uniform manner. In addition to that, Fig. 25 gives micrographs of those metal surface areas which were during the test partly covered by the ring seal, partly exposed to the nitric acid under heat transfer. No indication of crevice corrosion could be detected.

3.3 Comparison of Both Steels in Respect to their Corrosion Behavior

Fig. 26 compares the isothermal and heat transfer corrosion results of both steels as function of steel surface temperature. Remarkable differences in reaction rate and temperature dependence can not be derived from this figure. But our knowledge from all other findings and micrographs prove clearly that a successful long-term corrosion testing under heat transfer using "heated disk type" [1] devices could be performed in case of the Si-steel W.Nr. 1.4361 ESU only. Crevice corrosion disturbs the test results of those CrNi steels which are prown to pitting or crevice corrosion, like it did in case of W.Nr. 1.4306 n. One way out of this dilemma, already described by J. M. Harrison et al. [6], could be perhaps a reduction of the test duration, let say to 48 h, but that goes - because this test is too superficial - to the expenses of the test reproductility and/or the reliability of the results.

From the morphological point of view the attack on W.Nr. 1.4306 n reveals a heavy intergranular corrosion with all indications of a further increase of the reaction rate (gaps acting as crevices in the grain boundaries penetrating the metal bulk structure). In contrary, W.Nr. 1.4361 ESU develops when corroding a protective layer, uniform metal loss takes place at a constant rate, so without any tendency to an acceleration.

However, this behavior seems to be the easiest case of heat transfer corrosion and limited to corrosion testing in pure aceotropic nitric acid. If the acid is more diluted and contains oxidizing metal ions boiling at the heated steel surface can provoke local acid and metal ion concentrations and consequently accelerated corrosion (Fig. 27) [7].

4. SUMMARY

At first, a general survey on corrosion under heat transfer was given. Secondly, a suitable experimental method was chosen to obtain test results of stainless steel corrosion under simulated service conditions of a Purex process dissolver.

For that purposes two devices of the heated disc type were built, in which 5 mm steel plates were electrically heated on one side and exposed on the other side to concentrated nitric acid. The intended surface temperatures and temperature gradients could be realized and kept constant during test runs of up to 720 h duration without any major problem. In that way the austenitic CrNi steels DIN W.Nr. 1.4306 n (AISI type 304 L) and 1.4361, the latter in its electroslag remelted version, were tested in aceotropic nitric acid under heat transfer (metal surface temperatures: 120 - 150 °C, acid temperature: 110 °C).

Problems derived from crevice corrosion of the steel W.Nr. 1.4306 n in the gap between the ring seal and the metal surface. It limited the time of exposure under heat transfer for this steel to equal or less than 144 h.

The other steel W.Nr. 1.4361 ESU - self-protected by forming a uniform corrosion - resistant SiO_2 layer - did not show under heat transfer to the nitric acid any indication of crevice corrosion. The measured corrosion rate originates from uniform metal loss, which was controlled by the metal surface temperature. These findings are being presented in an Arrhenius plot, thus completing the results of Horn and Schoeller up to 150 °C. The given equation of the corrosion rate as function of temperature proved to be valid for our test results also.

The comparison of the tested steels in respect to their corrosion resistance goes in favour of the W.Nr. 1.4361 ESU. However, it must be emphasized that improved versions of the standard CrNi steel W.Nr. 1.4306 are available which show much lower corrosion rates than W.Nr. 1.4361 ESU, so that in case that a suitable steel is needed for aceotropic or lower concentration nitric acid services preference has to be given to the W.Nr. 1.4306 nitric acid grade (s) or to its electroslag remelted version (s ESU).

5. ACKNOWLEDGEMENTS

The authors give thanks to those coworkers who contributed to the general design of the test apparatus (Mr. R. Wolf) and the metallographic evaluation (H. Zimmermann and P. Graf).

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 (Late Paper, not included in printed conference proceedings)
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 J. Atom. Energy Soc., Japan 31 (1989) 1273

Designation	Chem. Composition	Results
PCTFE	Polychlortrifluorethylen	+
PFA	Perfluoralkoxy	+
FEP	Perfluorethylenpropylen	+
PTFE	Polytetrafluorethylen (Teflon)	+
Kalrez	Perfluorelastomer	о
	(Tetrafluorethylen/Perfluormethylvinylether)	
VITON	Fluorelastomer	-
Sigraflex	Graphit	_

Tab. 1:	Results of Qualitative Test Exposing Seals to Aceotropic Nitric Acid
	(120 °C), 100 - 165 h)

+ = resistant, o = reasonably resistant, - = non-resistant

r,)

<u>Tab 2:</u>	Chemical Composition of the Austenitic Stainless Steels which were
	tested in Aceotropic Nitric Acid under Heat Tranfer

DIN W.Nr	Producer Heat No.	Chem. Composition (wt-%)										
Designation		С	Si	Mn	Р	S	Cr	Mo	Ni	N	Co	Cu
<u>1.4306 n</u> Remanit 4306	TEW 681870	0,024	0,60	1,44	0,025	0,003	18,00	0,17	10,23	-	-	-
<u>1.4306 n</u> Remanit 4306	TEW 603650	0,022	0,42	1,59	0,033	0,003	18,23	0,30	10,26	-	-	-
<u>1.4361 ESU</u> A 610	VEW G 57128	0,007	4,10	0,50	0,018	0,001	17,52	0,13	14,75	0,013	-	-
<u>1.4361 ESU</u> A 610	VEW B 57377	0,007	4,10	0,74	0,020	0,001	17,28	0,15	15,50	0,016	-	-



Fig. 1 Parameters of Corrosion Tests in Nitric Acid and Nitric Acid Solutions

| 0 |



Fig. 2 Auflöser (Prinzip) / Dissolver (schematic)



115°C

Laboratory Simulation

130-160°C





 $\overline{\mathbf{N}}$

Fig. 4

Corrosion Testing under Heat Transfer: One Device in 4 Testing Positions



under Heat Transfer



Apparatus for Corrosion Testing under Heat Transfer and Acid Backward Removal



Isothermal Corrosion Testing of Solution Annealed 18 Cr 10 Ni LC Stainless Steel (W.Nr.1.4306n) in Aceotropic Nitric Acid (Huey-Test) (120°C, 15 × 48 = 720 h) 15



Fig. 8 Corrosion Testing under Heat Transfer of 18Cr 10Ni LC Stainless Steel (W. Nr. 1.4306 n) in Aceotropic Nitric Acid (Specimen : 120–150°C, Acid: 110°C, 3–15×48 = 144–720 h)

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Corrosion Testing under Heat Transfer of 18 Cr 10 Ni LC Stainless Steel (W.Nr.1.4306n) in Aceotropic Nitric Acid (Specimen: 120–150°C, Acid: 110°C, 3 × 48 = 144 h)



Fig. 10 Corrosion Testing under Heat Transfer of 18 Cr 10 Ni LC Stainless Steel (W.Nr.1.4306n) in Aceotropic Nitric Acid (Specimen: 120–150°C, Acid: 110°C, 3 × 48 = 144 h)



Corrosion Testing under Heat Transfer of 18 Cr 10 NiLC Stainless Steel (W.Nr.1.4306n) in Aceotropic Nitric Acid (Specimen: $120-150^{\circ}$ C, Acid: 110° C, $3 \times 48 = 144$ h)

— 19 —



 $50 \ \mu m$



Fig. 12

 $50\,\mu m$

Corrosion Testing under Heat Transfer of 18 Cr 10 Ni LC Stainless Steel (W.Nr. 1.4306 n) in Aceotropic Nitric Acid

Specimen: 120 °C, Acid: 110 °C, 240 h



 $50\,\mu m$



 $50\,\mu m$

Fig. 13

Corrosion Testing under Heat Transfer of 18 Cr 10 Ni LC Stainless Steel (W.Nr. 1.4306 n) in Aceotropic Nitric Acid

Specimen: 130 °C, Acid: 110 °C, 240 h



50 µm



Fig. 14

 $50\,\mu m$

Corrosion Testing under Heat Transfer of 18 Cr 10 Ni LC Stainless Steel (W.Nr. 1.4306 n) in Aceotropic Nitric Acid

Specimen: 140 °C, Acid: 110 °C, 240 h



 $50 \ \mu m$



 $50\,\mu m$

Fig. 15

Corrosion Testing under Heat Transfer of 18 Cr 10 Ni LC Stainless Steel (W.Nr. 1.4306 n) in Aceotropic Nitric Acid

Specimen: 150 °C, Acid: 110 °C, 144 h





Aceotropic Nitric Acid (Huey-Test) (120°C, 15 × 48 = 720 h)

-25



Fig. 18 X1CrNiSi18154, W.-Nr. 1.4361 und ESU-1.4361: Huey-Test mit kontinuierlicher Abführung der Korrosionsprodukte

Steel 1.4361 and ESR-1.4361: Modified Huey Test involving continuous removal of corrosion products Fig. 18 X1 CrNiSi 18154, W.-Nr. 1.4361: in azeotroper Salpetersäure 26

X1CrNiSi18154, steel 1.4361: corrosion resistance in azeotropic nitric acid

E.-M. Horn und K. Schoeller Werkstoffe und Korrosion 41, 97–112 (1990)



Oxidschichtaufbau auf einem 4,3% Si-haltigen CrNi-Stahl nach Salpetersäure-Korrosion

Oxide film on a 4,3 % Si-containing stainless steel after nitric acid corrosion (H. Diekmann et al., Z. Werkstofftechn. 9 (1978) 37-76)



В

Deckschichtaufbau auf W.Nr. 1.4361 unter Huey-Test-Bedingungen

Oxide Scale Formation of W.Nr. 1.4361 under Huey-Test Conditions (M. Maar-Stumm, KfK 4704 (March 1990))

Fig. 19



Fig. 20 Corrosion Testing under Heat Transfer of 17 Cr 14 Ni 4 Si Stainless Steel (W.Nr.1.4361 ESU) in Aceotropic Nitric Acid (Specimen: 120–150°C, Acid: 110°C, 720 h)



Fig. 21 Corrosion Testing under Heat Transfer of 17 Cr 14 Ni 4 Si Stainless Steel (W.Nr.1.4361) in Aceotropic Nitric Acid (Specimen: 120–150°C, Acid: 110°C, 720 h)



 $50\,\mu m$



Fig. 22

 $50 \ \mu m$

Corrosion Testing under Heat Transfer of 17 Cr 14 Ni 4 Si Stainless Steel (W.Nr. 1.4361 ESU) in Aceotropic Nitric Acid

Specimen: 120 °C, Acid: 110 °C, 720 h



 $50 \ \mu m$



 $50\,\mu m$

Fig. 23

Corrosion Testing under Heat Transfer of 17 Cr 14 Ni 4 Si Stainless Steel (W.Nr. 1.4361 ESU) in Aceotropic Nitric Acid

Specimen: 130 °C, Acid: 110 °C, 720 h



50 µm



Fig. 24

 $50\,\mu m$

Corrosion Testing under Heat Transfer of 17 Cr 14 Ni 4 Si Stainless Steel (W.Nr. 1.4361 ESU) in Aceotropic Nitric Acid

Specimen: 140 °C, Acid: 110 °C, 720 h



 $50 \ \mu m$



 $50\,\mu m$

Fig. 25

Corrosion Testing under Heat Transfer of 17 Cr 14 Ni 4 Si Stainless Steel (W.Nr. 1.4361 ESU) in Aceotropic Nitric Acid

Specimen: 150 °C, Acid: 110 °C, 696 h



Position of seal on metal

Acid e	exp	osed
metal	รบ	rface
┝──── ┥	50	μm



Position of seal on metal

Acid exposed metal surface ↓ 50 µm

Fig. 26

Corrosion Testing under Heat Transfer of 17 Cr 14 Ni 4 Si Stainless Steel (W.Nr.1.4361 ESU) in Aceotropic Nitric Acid (Specimen: 120/150°C, Acid: 110°C, 720/696 h)

No crevice corrosion below the plastic seal



Fig. 27 Corrosion Rates of CrNi Stainless Steels W.Nr. 1.4306/1.4361 in Aceotropic Nitric Acid under Isothermal and Heat Transfer Conditions

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Fig. 28

Relationship between metal surface temperature and corrosion rate of 310 Nb

Corrosion Behavior of Stainless Steels under Heat Transfer Condition in Simulated Solution of Dissolver for FBR Fuel Reprocessing

By Syotaro HAYASHI, Hisao OJIMA, [8] Katsuyuki KAWAGUCHI, Masatomo SHINOHARA and Takayuki KAWANO