〔環境材料工学科 〕

〔区 分 A〕

松英 達也

RELAXATION OF RESIDUAL STRESSES IN THIN FILMS INVESTIGATED USING SYNCHROTRON RADIATUION

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Advaces in x-ray Analysis・vol.52・233・2009年.
The crystal structure and residual stresses of TiN thin films deposited by arc ion plating (AIP) on a steel substrate were investigated using a synchrotron radiation system which emitted ultra-intense X-rays. In a previous study, the crystal structure of TiN films deposited by AIP was found to be strongly influenced by the bias voltage during deposition. However, for film thickness of approximately 200 nm, TiN films deposited under bias voltages of -100 V and 0 V had a preferred orientation of {110}. In the present study, the two-tilt method was used to evaluate the residual stresses in TiN films by measuring lattice strains in two directions determined by the crystal orientation. The TiN films deposited under a bias voltage of -100 V were found to have a compressive residual stress of -8.0 GPa. This was observed to decrease with increasing annealing temperature, and reached a value of -3.5 GPa at 800°C. On the other hand, TiN films deposited under a bias voltage of 0 V had a compressive residual stress of -6.5 GPa, which was reduced to -5.2 GPa following annealing at 800°C.
Keywords: TiN film, Arc ion plating, Crystal structure, Residual stress, Surface morphology, Synchrotron radiation

松英 達也

小・中学校理科および技術教員へのものづくりを通した実技研修会

松英達也[*1]、鎌田慶宣[*2]、谷口佳文[*1]、西井靖博[*3]、占部弘治[*4]、吉川貴士[*2]、松田雄二[*2]、
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平成21年度高専教育講演論文集･323-226・2009年
新居浜高専は平成18年度に文部科学省「現代的教育ニーズ取組支援プログラム（現代GP）」に「地域連携プロジェクト型ものづくり活動」を提案し、採択された。この活動は大きく分けて「ものづくり人材育成」と「まちづくり・地域の求心力向上」の２つのプロジェクトを柱としたものであり、それぞれに3つのプロジェクト活動を展開するよう組織化されている。教員の実技研修会は上記の「ものづくり人材育成・ものづくり教材開発プロジェクト」に組み込まれ、先の問題を解消し、より実践的な内容とするべく活動を行った。本論文では、平成18年度から平成20年度までの3年間の活動によって得られた成果について報告する。

朝日　太郎

Response characteristics of all-solid-state pH sensor using $Li_5YSi_4O_{12}$ glass

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A new type of all-solid-state pH sensor was investigated for the monitoring of pH in high temperature. The all-solid-state pH sensor consists of two half-cells: indicator electrode using the $Li_5YSi_4O_{12}$ glass and an Ag/AgCl reference electrode coated with Nafion film. A stable Nafion film was achieved by heat treating at 100 °C for 1 hour. The electromotive force (EMF) of the all-solid-state pH sensor decreased linearly with pH increase in water in accordance with the Nernst's equation. The all-solid-state pH sensor operated stably up to 80 °C. The sensitivity of the all-solid-state pH sensor against pH was high, and the EMF was also scarcely influenced by the presence of inorganic ions such as Li^+, Na^+ and Cl^-. It was practically confirmed by the pH titration test that the all-solid-state pH sensor behaved similarly to the commercial pH meter with the conventional glass electrode. In addition, the all-solid-state pH sensor showed same equivalence point both at high temperature and low temperature operations.

平澤　英之

小・中学校理科および技術教員へのものづくりを通した実技研修会

松英達也[*1]、鎌田慶宣[*1]、谷口佳文[*2]、西井靖博[*3]、占部弘治[*4]、吉川貴士[*2]、松田雄二[*2]、松友真哉[*4]、平澤英之[*1]、濱田　直[*5]

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平成21年度高専教育講演論文集、PP.223･225、2009

〔概要は前掲〕

平澤　英之

Heat Generation Ability in AC Magnetic Field of Nano $MgFe_2O_4$ Ferrite Powder Prepared by Bead Milling

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Proc. of the 6th International Symposium on Electromagnetic Processing of Materials, pp.189-192, 10/2009

The nano sized $MgFe_2O_4$-based ferrite powder having a heat generation ability in an AC magnetic field was prepared by bead milling and studied for thermal coagulation therapy applications. The crystal size and the particle size significantly decreased by bead milling. The heat generation ability in the AC magnetic filed improved with the milling time, i.e., a decrease in the crystal size. However, the heat generation ability decreased for excessive milled samples with crystal sizes

less than 5.5 nm. The highest heat ability (ΔT=34 ℃) in the AC magnetic field (370 kHz, 1.77 kA/m) was shown for the fine $MgFe_2O_4$ powder having an ca. 6 nm crystal size (the samples milled for 6-8 h using 0.1mmϕ beads). The heat generation of the samples was closely related to the hysteresis loss for the B-H magnetic property. The reason for the high heat generation properties of the samples milled for 6-8 h using 0.1mmϕ beads was ascribed to the increase in the hysteresis loss by formation of the single domain. Moreover, the improvement in the heating ability was obtained by calcination at low temperature of the bead-milled sample. In this case, the maximum heat generation (ΔT=41 ℃) ability was obtained for an ca. 11 nm crystal size sample prepared by the crystal growth during the sample calcination.

平澤 英之

Computer simulation gf heat generation ability in AC magnetic field for bneedle-type materials

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Proc. of the 6th International Symposium on Electromagnetic Processing of Materials, pp. 193-196, 10/2009

Considering the application for the novel ablation therapy of liver cancer, the heat generation ability of needle-type materials was studied in an AC magnetic field at 370kHz. The significant difference in the heating properties among the ferromagnetic Fe, Fe-based alloys and Ni was reasonably explained by the effect of the hysteresis loss. Moreover, that of the ferromagnetic Ni and non-magnetic Ti rod specimens was investigated using a computer simulation (JMAG Studio, ver.9.0) together with the experimental work. The Ni-rod specimen exhibited a markedly larger heat generation ability as compared to that of the Ti-rod specimen, resulting from the magnetic flux concentration at the surface and its vicinity. For these conducting materials, the reason for the temperature enhancement in the AC magnetic field was associated with the effect of eddy current loss. The heat generation abilities calculated from the computer simulation almost agreed with the experimentally obtained values. When the electrical resistivity is changed in the computer simulation, the peaks of the temperature and heat generation ability appeared around 0.40μΩm for the Ti-rod specimen. These results allowed us to conclude that the electrical resistivity plays a key role through the effects of the eddy current loss and magnetic penetration depth.

平澤 英之

High Heat Generation Ability under AC Magnetic Field for $Y_3Fe_5O_{12}$ Prepeared by Reverse Coprecipitation Method

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Proc. of the 6th International Symposium on Electromagnetic Processing of Materials, pp. 185-188, 10/2009

Magnetic materials having a high heat generation ability in an AC magnetic field have been studied for use in new thermal coagulation therapy for cancers. $Y_3Fe_5O_{12}$-based powder materials, i.e., the Gd-substituted $Y_{3-X}Gd_XFe_5O_{12}$ system, were synthesized by a reverse coprecipitation method for this

new therapy. The obtained precursors of homogeneous hydroxides using this method were calcined at 1000 ℃ - 1200 ℃ for the preparation of the $Y_3Fe_5O_{12}$-based oxide powder. A mixed cubic and orthorhombic phase was formed for the samples calcined below 1100 ℃ for all the examined X values. A single cubic phase was obtained for the samples heated at 1150 ℃ and higher. The maximum heat generation ability in an AC magnetic field (370 kHz, 1.77kA/m) was for the sample calcined at 1100℃. However, the heat ability was poor for the samples calcined at the lower and higher temperatures. The calcination temperature at around the phase transition between the two phases influenced the high heat generation ability. In particular, the particle growth with the formation of the cubic single phase acted to decrease the heat ability. The heat generation ability of the $Y_{3-X}Gd_XFe_5O_{12}$ system was almost the same for the X region from X=0 to X=2.5. However, the heat generation significantly decreased to zero for $Gd_3Fe_5O_{12}$ (X=3.0).

平澤 英之

Effect of crystal sizes on heat generation ability in AC magnetic field for $FeFe_2O_4$ ferrite powder prepared by bead milling

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For the treatment of cancerous tissues, the thermal coagulation techniques of radio frequency and microwave coagulation therapies have been applied as effective methods. Thermal coagulation can also be realized by application of an AC magnetic field from external coils to the tumors using powder type magnetic materials [1-3]. Moreover, the drug delivery system (DDS) using nano sized magnetic particles encapsulated in a liposome is applicable for this therapy.

Up to now, a fine magnetite ($FeFe_2O_4$) powder has been investigated as the candidate material for this type of therapy [1]. For the $FeFe_2O_4$, the heat generation ability in an AC magnetic field is influenced by the crystal size [2]. In our previous paper, it was reported that the $FeFe_2O_4$ prepared by coprecipitation method showed the maximum heating ability in an AC magnetic field when the crystal size was ca. 12 nm [3]. The heat generation in an AC magnetic field is strongly influenced by the hysteresis loss of the B-H magnetic property [2,3]. We have prepared the $FeFe_2O_4$ material by coprecipitation and reverse precipitation method [2]. However, the effect of the crystal size on the heat generation was not clear for these materials, because the particle size and the crystal size of the samples prepared by chemical processes might not be small enough to study these properties.

In this study, we investigated the heat generation ability in an AC magnetic field of fine $FeFe_2O_4$ powders physically prepared using a bead-milling method and calcinations in order to discuss the heating properties of the fine particles.

平澤 英之

Feasibility of chemohyperthermia with docetaxel-embedded magnetoliposomes as minimally invasive local treatment for cancer

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Hyperthermia is a minimally invasive approach to cancer treatment, but it is difficult to heat only the tumor without damaging surrounding tissue. To solve this problem, we studied the effectiveness of chemohyperthermia with docetaxel-embedded magnetoliposomes (DMLs) and an applied alternating current (AC) magnetic field. Human MKN45 gastric cancer cells were implanted in the hind limb of Balb-c/nu/nu mice. Various concentrations of docetaxel-embedded DMLs were injected into the tumors and exposed to an AC magnetic field (n 5 6, each). For comparison with hyperthermia alone, magnetite-loaded liposome (ML)-injected tumors were exposed to an AC magnetic field. Furthermore, the results of DML without AC treatment and docetaxel diluted into PBS with AC treatment were also compared (n 5 10, each). Tumor surface temperature was maintained between 42 and 43_C. Tumor volume was reduced in the DML group with a docetaxel concentration > 56.8lg/ml, while a docetaxel concentration > 568.5lg/ml was required for tumor reduction without hyperthermia. Statistically significant differences in tumor volume and survival rate were observed between the DML group exposed to the magnetic field and the other groups. The tumor disappeared in 3 mice in the DML group exposed to the magnetic field; 2 mice survived over 6 months after treatment, whereas all mice of the other groups died by 15 weeks. Histologically, hyperthermia with DML damaged tumor cells and DML diffused homogeneously. To the best of our knowledge, this is the first report to show that hyperthermia using chemotherapeutic agent-embedded magnetoliposomes has an anticancer effect.

〔区 分 C〕

松英 達也

The structure of Cr-N films deposited by arc ion plating investigated using synchrotron radiation

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The 10th International Symposium on Sputtering & Plasma Processes2009(ISSP2009)・531・2009年.

The structures of CrN films deposited by arc ion plating on steel substrates were investigated using a synchrotron radiation system that emits ultraintense X-rays. The CrN films were found to be mainly composed of {110} oriented CrN crystals, but they also had a small component of randomly oriented Cr_2N crystals. The CrN220 diffraction shifts to a high diffraction angle as the annealing temperature increases. In contrast, the peak position of the Cr_2N211 diffraction hardly changes with an increase in the annealing temperature up to 873K. The ratio of nitrogen and oxygen to chromium at the film surface and inside in the film was estimated by Auger electron spectroscopy. After annealing at 973K, the surface layer was oxidized, but the composition inside the CrN film (N/Cr=0.83) remained

unchanged.

松英 達也

RESIDUAL STRESSES OF Cr-N FILMS DEPOSITED BY ARC ION PLATING INVESTIGATED USING SYNCHROTRON RADIATION

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・The 5th International Conference on Mechanical Stress Evaluation by Neutrons and Synchrotron Radiation (MECASENSⅤ) Book of Abstracts・152・2009年.

The structures of Cr-N films deposited by arc ion plating on steel substrates were investigated using a synchrotron radiation system that emits ultraintense X-rays. The Cr-N films were found to be mainly composed of {110} oriented CrN crystals, but they also had a small component of randomly oriented Cr_2N crystals. The CrN220 diffraction shifts to a high diffraction angle as the annealing temperature increases. In contrast, the peak position of the Cr_2N211 diffraction hardly changes with an increase in the annealing temperature up to 873 K. The ratio of nitrogen and oxygen to chromium at the film surface and inside in the film was estimated by Auger electron spectroscopy. After annealing at 973 K, the surface layer was oxidized, but the composition inside the Cr-N films (N/Cr = 0.83) remained unchanged. The residual stress in a 1600-nm-thick as-deposited CrN layer was found to be -11.0GPa. The residual stresses of Cr-N films relaxed to thermal stress levels on annealing. However, the residual stress in the Cr_2N layer could not be evaluated.

松英 達也

Stress Estimation of Titanium Casting Alloy by x-ray Measurement Technique of Single Crystal

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・The 5th International Conference on Mechanical Stress Evaluation by Neutrons and Synchrotron Radiation (MECASENSⅤ) Book of Abstracts・83・2009年.

Residual stresses in titanium casting alloy were estimated by X-ray stress measurement technique. There are two problems in the condition of X-ray stress measurement. Firstly, the titanium casting alloy has the large crystal grains. These coarse grains were generated under solidification processes and those sizes are approximately 2 millimeter in this study. These coarse crystal grains interfere with an accurate stress measurement due to the unstable diffraction profile. This is because the existence of a sufficient number of isotropic crystal grains in the X-ray irradiation area are based on the X-ray diffraction theory. In this study, the stress measurement technique of single crystal materials was adopted for the solution of this fundamental problem. Because the coarse crystal grain was treated as a single crystal, the high intensity diffraction profiles were observed from a certain direction with investigations of crystal orientation. The problem with the coarse crystal

grain in titanium casting alloy were cleaned up by the employment of the single crystal measurement technique. Secondly, the results from this study show that the position of crystal grain within the X-ray irradiation area greatly influenced the residual stress values. Therefore, in the present paper the erasing method of this position effect was tried and discussed. Finally, the improvement of the accuracy of this method for the residual stress measurement in titanium casting alloy under the several bending stresses was confirmed. These results show that the erasing method in this study is an effective correction method.

松英 達也

Neutron Stress Measur6ment of Coarse Crystal Griin in Aluminum Casting AIloy

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・The 5th International Conference on Mechanical Stress Evaluation by Neutrons and Synchrotron Radiation (MECASENSⅤ) Book of Abstracts・133・2009年.

Internal stresses in aluminum casting alloy were measured by the neutron stress measurement method with the apparatus RESA in Japan Atomic Energy Agency (JAEA). In usual cases, coarse crystal grains are included in aluminum casting alloy. These coarse crystal grains make it extremely difficult to estimate the internal stresses by the neutron diffraction. The two problems arise because of the existence of the coarse crystal grains. The first problem is the production of an unstable diffraction profile in the necessary direction. The second is the edge effect which is generated by the overhang of the coarse crystal grains from the neutron irradiation area (gage volume). In this study, two kinds of new techniques used were proposed to resolve these problems. Firstly, the elastic theory based on the sin2ϕ method is shown. Diffraction peaks in several directions were found by use of the rocking curve method. Following that, the lattice spaces in each principal direction were calculated from these diffraction peaks using the elastic theory. Secondly, the distribution of edge effect around the gage volume was measured using a small bit of copper single crystal. From this result, the edge effect was canceled out by the modified measurement method which was done symmetrically. Finally, the aluminum casting sample which included coarse crystal grains was set to the tensile testing machine on RESA's measurement table and the applicability of these new techniques were confirmed experimentally.

朝日 太郎

部分安定化ジルコニアの低温熱劣化特性

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新居浜工業高等専門学校紀要、第46巻、p.29-32、2010.

1400、1500、1600℃で焼結した ZrO_2－x mol% Sm_2O_3、ZrO_2－x mol% Gd_2O_3、ZrO_2－x mol% Dy_2O_3、ZrO_2－x mol% Y_2O_3、ZrO_2－x mol% Yb_2O_3（x＝3、4、5）セラミックスの低温熱劣化特性について、X 線回折測定によって

検討したところ、以下のことがわかった。より低温で焼結したサンプルで、低温熱劣化が容易に起こらなかった。安定化剤量が多いサンプルで、低温熱劣化が容易に起こらなかった。よりイオン半径の大きな希土類元素で安定化されたサンプルで、低温熱劣化が容易に起こらなかった。

朝日　太郎

廃ガラスを用いた発泡性多孔質材料の作製

朝日太郎[*1]、中山享[*2]、大森大輔[*3]

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新居浜工業高等専門学校紀要、第46巻、p.47-50、2010.

廃ガラスの再利用化・資源化という観点から、水質浄化材や断熱材への利用を目指して、廃ガラス粉と発泡剤の焼成による発泡性多孔質材料の作製を試み、発泡剤の添加量や種類、焼成条件の変化による多孔性の検討を行った。特に、SiCを発泡助剤として使用した場合には、100μm付近のほぼ均一な細孔径を有する発泡体が生成することができ、SiCの添加量が多くなるほど発泡が進行し、比重が小さくなることが判明した。

〔区　分　E〕

新田　敦己

アナターゼ型酸化チタンを含む結晶化ガラスの作製

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日本セラミックス協会　2010年年会　2010年年会講演予稿集　2010年、P.35

Crystallization of B_2O_3-TiO_2-CaO system was investigated by the method of XRD, SEM, and EPMA. The glass was crystallized after heat-treating at 625℃ for 60 min in the atmosphere. The precipitated crystal in the glass was identified by the method of XRD. The crystal was only an anatase-type TiO_2. The crystals of both anatase and rutile type were deposited in the glass heat-treated at 700℃. But the amount of rutile-type was less than that of anatase-type. The above result shows that it is possible to produce a glass-ceramics with a photocatalyst function.

松英　達也

「たたら」製鉄法による炉体の設計と鋼の生成に関する研究～材料工学における「もの作り教育」～

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・第18回日本材料科学会四国支部講演大会(松山)・2009年6月

「たたら」とは、日本古来より伝わる製鉄技術であり、現代製鉄法と比べ生成効率が悪く連続操業にも向いていないが、良質の鋼を得ることができる。研究では、「たたら」製鉄による鋼の製造法の確立を目指し、主に炉体の構造および炉体材料と生成される鋼の組成について検討を行った。一方で、古式の製鉄法を学ぶことは現代の製鉄技術を理解するためには非常に有用であることから、このような活動を通じた材料技術者の育成にも役立つと考えられ、新居浜高専式たたら炉は携わった学生の努力の積み重ねにより完成したものである。このような研究活動が材料工学のもの作り教育の一助となり、多くの学生にもの作りの楽しさを感じる機会を与えられることを期待している。

松英 達也

The structure of Cr-N films deposited by arc ion plating investigated using synchrotron radiation

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・The 10th International Symposium on Sputtering & Plasma Processes2009(ISSP2009)・2009年７月

The structures of CrN films deposited by arc ion plating on steel substrates were investigated using a synchrotron radiation system that emits ultraintense X-rays. The CrN films were found to be mainly composed of {110} oriented CrN crystals, but they also had a small component of randomly oriented Cr_2N crystals. The CrN220 diffraction shifts to a high diffraction angle as the annealing temperature increases. In contrast, the peak position of the Cr_2N211 diffraction hardly changes with an increase in the annealing temperature up to 873K. The ratio of nitrogen and oxygen to chromium at the film surface and inside in the film was estimated by Auger electron spectroscopy. After annealing at 973K, the surface layer was oxidized, but the composition inside the CrN film (N/Cr=0.83) remained unchanged.

松英 達也

RESIDUAL STRESSES OF Cr-N FILMS DEPOSITED BY ARC ION PLATING INVESTIGATEDUSINGSYNCHROTRON RADIATION

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・The 5th International Conference on Mechanical Stress Evaluation by Neutrons and Synchrotron Radiation (MECASENSⅤ) ・2009年11月.

The structures of Cr-N films deposited by arc ion plating on steel substrates were investigated using a synchrotron radiation system that emits ultraintense X-rays. The Cr-N films were found to be mainly composed of {110} oriented CrN crystals, but they also had a small component of randomly oriented Cr_2N crystals. The CrN220 diffraction shifts to a high diffraction angle as the annealing temperature increases. In contrast, the peak position of the Cr_2N211 diffraction hardly changes with an increase in the annealing temperature up to 873 K. The ratio of nitrogen and oxygen to chromium at the film surface and inside in the film was estimated by Auger electron spectroscopy. After annealing at 973 K, the surface layer was oxidized, but the composition inside the Cr-N films (N/Cr = 0.83) remained unchanged. The residual stress in a 1600-nm-thick as-deposited CrN layer was found to be -11.0 GPa. The residual stresses of Cr-N films relaxed to thermal stress levels on annealing. However, the residual stress in the Cr_2N layer could not be evaluated.

松英 達也

Stress Estimation of Titanium Casting Alloy by x-ray Measurement Technique of Single Crystal

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・The 5th International Conference on Mechanical Stress Evaluation by Neutrons and Synchrotron Radiation (MECASENSV) ・2009年11月.
Residual stresses in titanium casting alloy were estimated by X-ray stress measurement technique. There are two problems in the condition of X-ray stress measurement. Firstly, the titanium casting alloy has the large crystal grains. These coarse grains were generated under solidification processes and those sizes are approximately 2 millimeter in this study. These coarse crystal grains interfere with an accurate stress measurement due to the unstable diffraction profile. This is because the existence of a sufficient number of isotropic crystal grains in the X-ray irradiation area are based on the X-ray diffraction theory. In this study, the stress measurement technique of single crystal materials was adopted for the solution of this fundamental problem. Because the coarse crystal grain was treated as a single crystal, the high intensity diffraction profiles were observed from a certain direction with investigations of crystal orientation. The problem with the coarse crystal grain in titanium casting alloy were cleaned up by the employment of the single crystal measurement technique. Secondly, the results from this study show that the position of crystal grain within the X-ray irradiation area greatly influenced the residual stress values. Therefore, in the present paper the erasing method of this position effect was tried and discussed. Finally, the improvement of the accuracy of this method for the residual stress measurement in titanium casting alloy under the several bending stresses was confirmed. These results show that the erasing method in this study is an effective correction method.

松英 達也

Neutron Stress Measurement of Coarse Crystal Grain in Aluminum Casting Alloy

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・The 5th International Conference on Mechanical Stress Evaluation by Neutrons and Synchrotron Radiation (MECASENSV) ・2009年11月.
Internal stresses in aluminum casting alloy were measured by the neutron stress measurement method with the apparatus RESA in Japan Atomic Energy Agency (JAEA). In usual cases, coarse crystal grains are included in aluminum casting alloy. These coarse crystal grains make it extremely difficult to estimate the internal stresses by the neutron diffraction. The two problems arise because of the existence of the coarse crystal grains. The first problem is the production of an unstable diffraction profile in the necessary direction. The second is the edge effect which is generated by the overhang of the coarse crystal grains from the neutron irradiation area (gage volume). In this study, two kinds of new techniques used were proposed to resolve these problems. Firstly, the elastic theory based on the $\sin 2\phi$ method

is shown. Diffraction peaks in several directions were found by use of the rocking curve method. Following that, the lattice spaces in each principal direction were calculated from these diffraction peaks using the elastic theory. Secondly, the distribution of edge effect around the gage volume was measured using a small bit of copper single crystal. From this result, the edge effect was canceled out by the modified measurement method which was done symmetrically. Finally, the aluminum casting sample which included coarse crystal grains was set to the tensile testing machine on RESA's measurement table and the applicability of these new techniques were confirmed experimentally.

松英　達也

小・中学校理科および技術教員へのものづくりを通した実技研修会

松英達也[*1]、鎌田慶宣[*2]、谷口佳文[*2]、西井靖博[*3]、占部弘治[*4]、吉川貴士[*2]、松田雄二[*2]、松友真哉[*4]、平澤英之[*1]、廣田　直[*5]

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平成21年度独立行政法人国立高等専門学校機構主催教育教員研究集会（豊田市）・2009年8月

〔概要は前掲〕

朝日　太郎

Na_2O-Y_2O_3-SiO_2系ガラスの電気特性と耐水性

朝日太郎[*1]、丸岡　研[*2]、清野　肇[*3]、中山　亨[*4]

[*1]新居浜工業高等専門学校環境材料工学科、[*2]新居浜工業高等専門学校専攻科生物応用化学専攻、[*3]北海道大学、[*4]新居浜工業高等専門学校生物応用化学科

第22回日本セラミックス協会秋季シンポジウム　2009年

本全固体型 Na^+イオンセンサの検知極材料などへの利用を目的として、Na_2O-Y_2O_3-SiO_2系ガラスの組成とガラス化状態、熱的特性、^{23}Na MAS-NMR による構造、Na^+イオン導電率、水への Na 浸出率について検討を行った。Na^+イオン導電率は、導電種である Na 含有量が増大するにつれて高くなり、Na 含有量が一定の場合では$[Y_2O_3]/[SiO_2]$比に関係なく Na^+イオン導電性はほとんど変化しなかった。また、水への Na 浸出率は、Na 含有量が増大するにつれて高くなり、Na 含有量が一定の場合では$[Y_2O_3]/[SiO_2]$比が大きくなると低下した。

朝日　太郎

$Li_5YSi_4O_{12}$ガラスを用いた全固体型 pH センサの応答特性

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第 22 回日本セラミックス協会秋季シンポジウム　2009 年

全固体型 pH センサの 25～80 ℃の温度域における水溶液の pH モニタリングについて検討した。本全固体型 pH センサは、$Li_5YSi_4O_{12}$ ガラスを用いた pH 検知極と Nafion 膜をコーティングした金属銀/塩化銀（Ag/AgCl）参照極の 2 つの電極からできている。その起電力は、水溶液中の pH 増加に伴い Nernst 式に従って直線的に減少した。また、その起電力は、ナトリウムイオン（Na^+）や塩化物イオン（Cl^-）のような無機イオンの存在にほとんど影響を受けなかった。本全固体型 pH センサが、ガラス電極を用いた市販の

pH メータとよく似た挙動を示すことが pH 滴定によって確かめられた。加えて、この全固体型 pH センサは 80℃でも安定作動した。

朝日　太郎

リン酸ジルコニウムによる Cs 固定化

近藤正太[*1]、高橋麻衣子[*2]、朝日太郎[*3]、中山　亨[*2]

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第 16 回　ヤングセラミスト・ミーティング in 中四国　2009 年

二次元層状構造を持つプロトン型リン酸ジルコニウム（α-$Zr(HPO_4)_2 \cdot H_2O$）を用いた Cs イオン固定化及びその Cs 耐浸出特性について、先に報告している三次元網目構造を持つプロトン型リン酸ジルコニウム（$HZr_2(PO_4)_3$）を用いた場合と比較し検討した。

朝日　太郎

ナトリウムーケイ酸ガラスの耐水性への添加元素の影響

丸岡　研[*1]、朝日太郎[*2]、清野　肇[*3]、中山　亨[*4]

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第 16 回　ヤングセラミスト・ミーティング in 中四国　2009 年

Na_2O-SiO_2 ガラスの耐水性向上を目的として、添加元素の影響を調べた。さらに、ガラス転移温度、Na^+ イオン導電性及び ^{23}Na MAS -NMR による Na 周囲の構造についても検討を行った。

朝日　太郎

希土類ケイ酸塩を用いた全固体型イオン電極の応答に及ぼす電解処理効果

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全固体型 Na^+イオン電極（Pt ワイヤー｜Ag ペースト｜$Na_5YSi_4O_{12}$ ガラス板）の起電力安定性及び起電力再現性の改善を目指して、$Na_5YSi_4O_{12}$ を負極とし Pt を正極として NaCl 水溶液中での電解処理効果について検討した。その電解処理により、電極の応答特性向上が認められた。その原因は、電極界面付近で起こるイオン交換による $Na_5YSi_4O_{12}$ 内のイオン活量が溶液中のイオン濃度の変化によってほとんど変化せず一定値を保つようになったためと考えられる。

平澤　英之

TiAl 合金の高温酸化

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日本材料科学会四国支部　第 18 回講演大会　2009 年 6 月

TiAl 合金は高温における強度・耐クリープ性について優れた軽量耐熱材料であり、タービンブレードや自動車用ターボチャージャーローターなどに用いられている。しかし、近年の航空機、自動車等の高速化により、それらの耐熱材料においては益々過酷な高温環境となり、さらに高温での耐酸化性の良好な超耐熱材料の開発が望まれている。

TiAl を酸化させた場合、生成する酸化物は TiO, Ti_2O_3, TiO_2, Al_2O_3, Al_2O, AlO, Al_2TiO_5 などが考えられて

いるが、実際の TiAl 相の酸化において酸化物の形態とそれらの形成過程を明らかにし、さらに酸化反応全体を支配しているものは何かという観点から、酸化反応における構成元素の拡散に関する知見も必要である。

本研究は、TiAl 基超耐熱合金開発のための基礎研究であり、その第一段階として TiAl 合金の耐酸化特性に関するデータを得るため、大気中で 1123K～1273K の高温で酸化させた場合の酸化被膜の成長などを化学天秤による重量変化、SEM および EPMA 観察などで調べ、本合金の酸化挙動とその特徴を明らかにするものである。

平澤 英之

物理的手法によるマグネタイトの粒子径制御と交流磁場中での発熱特性

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日本セラミックス協会　第 22 回秋季シンポジウム　H21 年 9 月

The nano-sized $FeFe_2O_4$ ferrite powder having a heat generation ability in an AC magnetic field was prepared by beads milling. The highest heat generation ability (ΔT=26 ℃) was obtained ca. 4.7nm in crystal size. Moreover, the improvement in the heating ability was obtained by calcination in Ar atmosphere at low temperature of the bead-milled sample.

平澤 英之

ガーネット系 $Y_{3-X}Gd_XFe_5O_{12}$ 型フェライトの交流磁場中での発熱特性

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日本セラミックス協会　第 22 回秋季シンポジウム　H21 年 9 月

We prepared the $Y_{3-X}Gd_XFe_5O_{12}$ ferrite system using a reverse coprecipitation method. The maximum heat generation ability (ΔT) was obtained for the samples calcined at around 1100℃. However, the $Gd_3Fe_5O_{12}$ (X=3.0) has no heat generation ability in AC magnetic field. The calcined temperature of the maximum ΔT value would depend on the crystal growth with the phase change between cubic and orthorhombic phases.

平澤 英之

様々な化学的作製法により作製した Mg1-XCaXFe2O4 系フェライトの交流磁場中での発熱特性

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日本セラミックス協会　第 22 回秋季シンポジウム　H21 年 9 月

We prepared the $Mg_{1-X}Ca_XFe_2O_4$ ferrite system using two methods of ethylene glycol method and reverse coprecipitation method. The maximum heat generation ability (ΔT) was obtained for the samples calcinedat around 700℃ and 800℃ for the two methods, respectively. The calcined temperature of the maximum ΔT value would depend on the crystal distribution with the phase change between cubic and orthorhombic phases.

平澤 英之

癌の交流磁場焼灼療法への応用を目的としたフェライト材料の設計

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日本化学会西日本大会　H21 年 11 月

交流磁場中で著しく発熱する材料は、癌の新しい焼灼療法（図１）への応用を目的として、発熱特性の優れたものが切望されている。これは、磁性材料を腫瘍部に留置し、交流磁場により焼灼するものである。そして、磁性材料を患部に留置する方法として３種が考えられており、

(1) 針状磁性材料を患部に穿刺し、焼灼後に抜き取る方法

(2) 球状磁性材料(20～30μm)をカテーテルにより患部の血管に塞栓させる方法

(3) 微粒子磁性材料(50nm 以下)を抗体付きリポソームに包埋しカテーテルを用いて選択的に患部に堆積させる究極の方法

である。この交流磁場焼灼法は、効果的に患部にダメージを与え、さらにヒートショックプロテイン (HSP) の発現により正常細胞の免疫機能を活性化させる。本発表の研究は(2)(3)で用いるフェライト材料の研究であり、多くの研究グループが FeFe204 を用いて研究を行ってきているが、我々は FeFe204 も含めた発熱能力の優れた材料の検索及び研究を行ってきている。そして、これまでに様々な材料を候補として報告してきている、この作製方法としては共沈法や高分子化法などによる化学的作製法による微粒子化が主であり、一方ビーズミルによるナノ微粒子への物理的粉砕も有効である。ここではそれらの材料の設計指針について述べる。

平澤　英之

癌治療への応用を目的としたガーネット系$Y_3Fe_5O_{12}$フェライトのビーズミル粉砕による微粒子化と交流磁場申での発熱特性

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日本セラミックス協会2010年年会　H22年3月

Fine $Y_3Fe_5O_{12}$ ferrite powder was prepared using bead-milling. Although the commercial $Y_3Fe_5O_{12}$ dose not show heat generation ability (ΔT=0℃) at al., the heat generation ability was strongly improved by the bead-milling. The maximum heat temperature (ΔT=60 ℃) in an AC magnetic field was obtained for the sample having 16 nm in crystal size.

平澤　英之

ガーネット系 $Y_3Fe_5O_{12}$ を基本とするフェライトの交流磁場中での発熱特性

森谷健史[*1]、青野宏通[*1]、猶原　隆[*1]、前原常弘[*2]、渡部祐司[*3]、平澤英之[*4]

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日本セラミックス協会2010年年会　H22年3月

We prepared the $Y_3Fe_5O_{12}$-based ferrite, i.e., $Y_{3-X}Gd_XFe_5O_{12}$ system using a reverse coprecipitation method. The maximum heat generation ability (ΔT) was obtained for the samples calcined at 1100 ℃. The calcined temperature of the maximum ΔT value would depend on the crystal growth with the phase change between cubic and orthorhombic phases. However, the $Gd_3Fe_5O_{12}$ (X=3.0) has no heat generation ability in AC magnetic field.