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<報告>

2022 年度 材料技術研究協会討論会報告

<EDITORIAL NOTE>

Preparation and polymerization of pentaethoxydisiloxane

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Abstract

We previously reported the synthesis of pentaethoxydisiloxane (PEDS) by the oxidation of triethoxysilane (TES) with oxygen gas in the presence of a Rh catalyst. However, the isolate yield of PEDS was low (< 7%). Herein, we report a PEDS yield of 62% using a new synthetic method and the characterization of the PEDS polymer by nuclear magnetic resonance (NMR) and Fourier transform infrared (FTIR) spectroscopy. The PEDS polymer was synthesized by hydrolysis–condensation of PEDS, and this polymer was composed of linear and branched chains and was the end group of the Q¹ unit.

Keywords : Hetero-disiloxane, Polymerization, NMR, FTIR

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1. Introduction

Pentaethoxydisiloxane (PEDS) is a heterosiloxane having a hydrido group and can be substituted by addition reaction with olefins ¹⁾. We previously reported that PEDS was synthesized by the oxidation of triethoxysilane (TES) in the presence of Rh(PPh₃)₃Cl catalyst and activated molecular sieves with oxygen gas bubbling ²⁾. This reaction proceeded via the formation of silanol (Si– OH) via the oxidation of the Si–H bond, followed by condensation between Si–OH and Si–OEt. However, this condensation reaction produces ethanol, and Si– OEt is formed by the reaction between Si–H and ethanol (Figure 1). Activated molecular sieves are necessary to remove ethanol.



Figure 1 Scheme for oxidation of triethoxysilane. Numbers in parentheses indicate the GC area ratio.

We found that the side reactions could be suppressed when the adsorption rate of the molecular sieves was faster than the rate of ethoxylation ³). However, this reaction is an inefficient and inappropriate synthetic method for PEDS because the isolated yield of PEDS was 7%. According to the reaction mechanism, PEDS can be synthesized via the reaction of isolated triethoxysilanol (TESOL) with TES. Herein, we report a new synthetic method for PEDS. Moreover, we studied the PEDS polymerization and its structure, which could not be polymerized due to insufficient amount of PEDS synthesized by the previous method.

2. Experimental section

2.1 Materials

Toluene and tetrahydrofuran (THF) were purified using standard methods and stored in activated molecular sieves. Triethoxysilane (TES), tetraethoxysilane (TEOS), chlorotrimethylsilane (TMSCl), and thionyl chloride (SOCl₂) were purchased from Tokyo Chemical Industry Co. Ltd. (Japan), and TES and TEOS were purified by distillation. Ammonium carbonate and N,Ndimethylformamide (DMF) were purchased from FUJIFILM Wako Pure Chemical Corporation (Japan). Water was purified using a PURELAB Flex 5 water purification system (ELGA, UK) with a resistivity of ~15 M Ω ·cm.

2.2 Measurements

GC analysis was carried out on a GC-390 (GL Science, Japan) packed with an SE-30 capillary column (Agilent, USA) and a TCD detector. Helium was used as the carrier gas. The column temperature was programmed as follows: injection temperature at 250 °C, isothermal state at 80 °C for 2 min, then heating up to 200 °C at rate of 10 °C min⁻¹, followed by heating up to 280 °C at rate of 20 °C min⁻¹, and held at the final temperature of 280 °C for 2 min. Nuclear magnetic resonance (NMR) spectra were recorded on a JEOL Resonance JNM-ECP 300 spectrometer (JEOL, Japan; ¹H at 300 MHz, ¹³C at 75 MHz, and ²⁹Si at 60 MHz). Chemical shifts were reported in ppm relative to chloroform-d (CDCl₃) as the internal standard (for ¹H:7.26 ppm and ¹³C:77.16 ppm in residual chloroform) and tetramethylsilane as the internal standard (for ²⁹Si:0.00 ppm). For the ²⁹Si NMR spectra, Cr(acac)₃, as a paramagnetic relaxation agent, was added to the samples. Gel permeation chromatography (GPC) was performed

using an HPLC system (LC-6AD, Shimadzu, Japan) attached to a PLgel 5 μ m Mixed-D column. THF was used as the eluent (1 mL min⁻¹). RID-10A was used as the detector at 40 °C. The molecular weights were calculated based on polystyrene standards. Fourier-transform infrared (FTIR) spectra were recorded on an FT-IR spectrophotometer (FT/IR-6100, JASCO, Japan) using the neat method, in which the sample was sandwiched between two KBr crystal disks.

2.3 Preparation of triethoxysilanol (TESOL)

TESOL was prepared according to literatures ^{4,5)}. TEOS (26 g, 0.13 mol) was added to a mixture of SOCl₂ (14.9 g, 0.13 mol) and DMF (0.13 mL), and then stirred for 4 h. Chlorotriethoxysilane was separated by distillation under reduced pressure (70-72 °C/36 mmHg) and obtained as colorless liquid (yield 59%, GC purity 82%). A solution of chlorotriethoxysilane (9.9 g, 0.05 mol) and 100 mL THF was added, while stirring under an ice bath, to a suspension of water (4.5 g, 0.25 mol), (NH₄)₂CO₃ (28.5 g, 0.25 mol), and THF (250 mL). After stirring for 1 h, the precipitate was filtered, and the filtrate was dried over Na₂SO₄. Thereafter, THF was evaporated in ice bath under reduced pressure, and TESOL was separated by distillation under reduced pressure (61 °C/1 mmHg). TESOL was a colorless liquid (yield, 62%; GC purity, 82%), but TEOS was contaminated (12%).

2.4 Synthesis of PEDS

TESOL (1.1 g, 6.1 mmol) was added using a dropping funnel to a solution of TES (1.0 g, 6.1 mmol) and toluene (3 mL) and stirred at 100 °C. After 4 hours, PEDS was isolated by distillation under reduced pressure (104 °C/11 mmHg) to obtain

a colorless liquid (yield: 62%).

¹H NMR: δ = 1.24 (t, *J* = 7.0 Hz, 9H, CH₃ in Q unit), 1.25 (t, *J* = 6.9 Hz, 6H, CH₃ in T unit), 3.86 (q, *J* = 7.0 Hz, 6H, CH₂ in Q unit), 3.87 (q, *J* = 6.9 Hz, 4H, CH₂ in T unit), 4.35 (s, 1H, Si–H). ¹³C NMR: δ = 18.05 (CH₃), 18.17 (CH₃), 58.27 (CH₂), 59.23 (CH₂). ²⁹Si NMR: δ = -67.7, -88.4.

2.5 Polymerization of PEDS

PEDS (5.0 g, 17 mmol) and ethanol (25 mL) were placed in a 100 mL four-necked flask equipped with nitrogen inlet and outlet tubes and a mechanical stirrer. The mixture was stirred at 200 rpm and then cooled in an ice bath for 20 min. Thereafter, conc. HCl (aq) (0.28 mL), water (0.09 mL), and ethanol (5 mL) were added (HCl/PEDS=0.2, H₂O/PEDS=1.0). The resulting mixture was stirred for 10 min in an ice bath and then for 10 min at 22±3 °C. The hydrolysis– condensation reaction was carried out at 80 °C for 3 h under a nitrogen flow of 200 mL min⁻¹. The PEDS polymer was obtained as a colorless and viscous liquid (3.2 g). The average molecular weight (M_w) and polydispersity index were 24,000 and 6.8, respectively, as calculated by GPC.

2.6 Hydrolysis of PEDS followed by addition of TMSCl

PEDS (1.5 g, 5 mmol) was mixed in ethanol (7.5 mL) in an ice bath for 20 min. Then, conc. HCl (aq) (0.08 mL), water (0.03 mL), and ethanol (1.5 mL) were added (HCl/PEDS=0.2, H₂O/PEDS=1.0). The resulting mixture was stirred in an ice bath for 10 min, followed by the addition of TMSCl (0.64 mL). After stirring, the solvent was evaporated, and a colorless liquid was obtained.



Figure 2 GC traces of the reaction solution of (a) TESOL with TES and (b) TESOL with TEOS at 100 °C for 4 h

3. Results and discussion

3.1 Synthesis of PEDS by the reaction of TESOL with TES

A GC chart of the TES and TESOL reaction solutions is shown in Figure 2a. TEOS was observed because TESOL was contaminated with TEOS; therefore, TES was not completely consumed. However, strong peaks corresponding to PEDS and ethanol were observed, indicating that the reaction between TESOL and TES proceeded under neutral conditions. In contrast, Figure 2b shows the GC chart of the reaction solution of TEOS with TESOL, and the intensities of ethanol and hexaethoxydisiloxane (HEDS) were very weak, indicating that little reaction occurred between TESOL and TEOS. TESOL could not be observed because it may have been adsorbed onto the SE-30 capillary column. As a result, TES is easily attacked by the silanol group and siloxane linkages can be formed even under neutral conditions because of the low steric hindrance of Si-H⁶).

3.2 Characterization of PEDS polymer

The PEDS polymer was prepared by the hydrolysis-condensation reaction of PEDS under nitrogen gas flow. Nitrogen gas was introduced to remove the hydrochloric acid catalyst and accelerate the condensation reaction by evaporating the solvent ^{5,7,8)}. The ²⁹Si NMR spectra of PEDS and PEDS polymers are shown in Figure 3. PEDS displays two signals at -67.7 and -88.4 ppm due to T¹ unit (HSi(OEt)2OSi(OEt)3) and O^1 unit (HSi(OEt)₂OSi(OEt)₃), respectively. PEDS polymer was confirmed by six obvious signals (-68.0, -77.2,-86.4, -88.9, -96.3, and -104 ppm) and four shoulder signals (-76.0, -84.6, -95.4, and -103 ppm). Because the cyclic form generally has a lower magnetic field than linear and branched chains $^{9)}$, the obvious signals were attributed to linear and branched chains, and shoulder signals were assigned to the cyclic form. However, the intensity of the shoulder signals was weak, indicating that only a small amount of cyclic form was present. The T¹ and Q¹ units of the PEDS polymer shifted to a slightly higher magnetic field than those of PEDS. This indicates that the T¹ and Q¹ units are the end groups of the PEDS polymer. The intensity of the T¹ unit was very weak; hence, the end group of the PEDS polymer was mainly the Q¹ unit. These attributes are summarized in Table 1.

The FTIR spectra of the PEDS and PEDS polymers are shown in Figure 4, and the absorption bands were attributed with reference to the literature ^{10,11} and are summarized in Table 2. The position of vSi–H in the PEDS polymer shifted to a higher

wavenumber (from 2212 to 2240 cm⁻¹) than that in pristine PEDS, indicating that the condensation reaction proceeded ^{12,13}.

Figure 3 ²⁹Si NMR spectra of PEDS and PEDS polymer

Table 1 Assignment for ²⁹Si NMR spectra of PEDSs

PEDS	PEDS polymer	Assignment
-67.7	-68.0	T^1
	-76.0	Cyclic T ²
	-77.2	Linear T ²
	-84.6	Cyclic T ³
	-86.4	Branched T ³
-88.4	-88.9	Q^1
	-95.4	Cyclic Q ²
	-96.3	Linear Q ²
	-103.0	Cyclic Q ³
	-104.0	Branched Q ³

Figure 4 FTIR spectra of PEDS and PEDS polymer

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Table 2 Assignment for FTIR spectra of PEDSs

PEDS	PEDS polymer	Assignment ^a
2979, 2931, 2890	2982, 2931, 2896	$\upsilon CH_2, \upsilon CH_3$
2212	2240	υSi–H
1487–1296	1487–1296	$\delta CH_2, \delta CH_3$
1170	1167	ρCH ₃
	1150-1050	vSi-O-Si
1103, 1081	1103, 1081	υC–Ο
970	970	vCC
882	885	ωSi–H
	853	vSi-O-Si
796	793	vSi-O-Si
758	755	$ ho CH_2$
710, 634, 612		
482	479	δSi-O-Si

^a v: stretching, δ : scissoring, ω : wagging, ρ : rocking

The shape of vSi-O-Si at 1150-1050 cm⁻¹ indicated that the PEDS polymer was not comprised of ladder-like, cage-like, or strained cyclic structures ^{14–16}, that is, the PEDS polymer was composed of linear, branched, and cyclic structures. However, the absorption band at ~550 cm⁻¹ due to the cyclotetrasiloxane moiety ¹⁷) was very weak. Thus, the structure of the PEDS polymer consists mainly of linear and branched chains, which is consistent with the ²⁹Si NMR results. From the results of ²⁹Si NMR and FTIR, the PEDS polymer was composed of mainly linear and branched chains and had an end group of Q1 unit. Si-H can be modified by hydrosilylation ¹⁾, alkoxylation ¹⁸⁻²⁰⁾, hydroxylation ²¹⁾, and siloxylation ^{22,23)}; hence, PEDS polymers can be used as precursor polymers for many functional siloxane polymers and materials, such as mesoporous silica ^{24–26}.

3.3 Hydrolysis of PEDS

To measure the initial stage of PEDS hydrolysis reaction, PEDS was hydrolyzed at ~0 °C for 10 min and silanol was capped with TMSCl. Figure 5 shows

the ²⁹Si NMR spectra of the hydrolysate. Two signals of the T² unit at ~ 75 ppm and two signals of the Q² unit at ~ 96 ppm were confirmed because the signals of trimethylsilyl-capped Tⁿ and Qⁿ units appeared at a lower magnetic field than that of the condensation of T–T or T–Q ²⁷⁾. The T¹ unit disappeared, and the Q¹ unit remained, indicating that the hydrolysis and condensation rates of the T¹ unit were faster than those of the Q¹ unit. The T unit is mainly transformed into the T³ unit.

The signal pattern of the initial stage of hydrolysis of the PEDS differed from that of the PEDS polymer. We consider that the Si-O-Si network of the PEDS polymer was rearranged during the condensation reaction because Si-O-Si bonding is cleaved under acidic conditions ²⁸⁾. Therefore, we believe that condensation reactions using formic acid can be used to obtain polymer-maintained PEDS backbones because formic acid can be easily transformed into formate compounds ^{29,30}. In the future, we will investigate this hypothesis by performing hydrolysis-condensation of PEDS using formic acid. The changes in the properties due to the polymer structure will also be investigated.

Figure 5 ²⁹Si NMR spectra of PEDS hydrolysis followed by addition of TMSC1. The "cap." means capped with trimethylsilane, and the "cond." means condensation between PEDSs.

4. Summary

We report a new method for the synthesis of PEDS via the reaction of TES with TESOL. In the previous method using the oxidation reaction of TES in the presence of the Rh catalyst, the isolated yield was 7%. In comparison, the isolated yield of PEDS by the new synthetic method was 62%, which was approximately nine times greater than that obtained by the previous method. The PEDS polymer was prepared by the hydrolysis-condensation reaction of PEDS under acidic conditions. This polymer is composed of both linear and branched chains. However, the PEDS backbone in this polymer is likely to be partially disintegrated, which is considered to perform a condensation reaction under acidic conditions. Therefore, it may be possible to synthesize polymers that retain the PEDS backbone by performing condensation reactions under neutral conditions.

Conflict of interest

The authors declare no competing interests.

CRediT authorship contribution statement

Iori Abe: Writing – original draft, visualization, investigation; Yohei Sato: Writing – original draft; Ryohei Hayami: Writing – original draft, visualization, investigation; Kazuki Yamamoto: Supervision; Satoru Tsukada: Supervision; Takahiro Gunji: Writing – review and editing, conceptualization, supervision.

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2022 年度 材料技術研究協会討論会報告

討論会委員会委員長	酒井	秀樹	(東京理科大学)
討論会実行委員長	羽田	宣弘	(環太平洋大学)

2022年12月1日(木),2日(金)の2日間,2022 年度 材料技術研究協会討論会が開催されました。年末 のお忙しい時期に討論会で発表していただいた方々, 参加いただいた方々,また討論会運営にご支援をいた だいた方々に心よりお礼申し上げます。

本年度の討論会をどのような形式で実施するかにつ いては,討論会委員会,理事会の中でも多くの議論が 行われましたが,最終的に対面を基本として一部来場 が困難な方に対してオンラインを併用する形をとるこ とになりました。結果として,東京理科大学神楽坂キ ャンパス(森戸記念館)に久しぶりに多くの方に集っ ていただき盛会となりました。

討論会1日目(12月1日)

討論会委員長・副委員長による開会挨拶の後,9時 30分からA会場,B会場の2セッションに分かれて口 頭講演が始まりました。久しぶりの対面での発表とな り,活発な質疑が行われました。また,同時にZoomを 用いてオンラインでも講演が発信されました。

その後 10 時 40 分からは,本討論会はじめての試み として,「環境・エネルギー研究部会シンポジウム」が 開催されました。シンポジウムの冒頭では,以下のお 二人の先生による特別講演が行われました。

・高見則雄先生(東芝 研究開発センター)

「チタン酸化物系負極を用いた大型二次電池の技術と 展望」

·嘉藤 徹先生 (産業技術総合研究所)

「高温水蒸気電解による水素製造」

当該分野のフロントランナーでいらっしゃる先生方 のご講演は,関連分野のみならず,他分野の聴講者に とってもインパクトがある素晴らしい講演でした。

昼食を挟んで午後からは、小浦 節子教授(千葉工 業大学、本会会長(当時))による総合講演が、「これ までの研究生活、これからの Well Being」の演題で行 われました。小浦先生がこれまで取り組んでこられた イオン液体を用いたアルミニウムの電析、ウルトラフ ァインバブル水のご研究に加えて、「これからの研究開 発においては、人々の Well Being ために必要なテーマ を考えていくことが重要である」とのメッセージは、 これから研究を展開される若い研究者、学生の皆さん には大変勉強になったのではないかと思います

総合講演に引き続き、表面改質部会の特別セッショ ンが開催され、以下4件の依頼講演が行われました。 ・「アクリル粘着剤の設計技術と加飾フィルム用途へ の応用」(綜研化学株式会社)後藤成吾先生

•「動的表面張力入門」

(協和界面科学株式会社) 磯貝洋幸 先生

・「UV/ozone表面改質を施した細胞培養基材による多 能性幹細胞の高効率培養」(慶應大学理工学部機械工学 科) 宮田昌悟 先生

・「電池材料の開発を指向した金属アルコキシド法に よる粒子へのコーティング」(北見工業大学工学部)大 野智也,平井慈人, Jeevan Kumar Padarti,松田 剛 各 先生

その後は、再びパラレルセッションとなり、A 会場 では、「環境・エネルギー研究部会シンポジウム」の一 般講演が行われ、環境・エネルギーに関する広範な分 野からの話題提供が行われました。並行して、B 会場 では、主に生体材料分野の一般ロ頭講演が実施され、 活発な質疑応答が行われました。

<u>討論会2日目(12月2日)</u>

討論会2日目も、朝のセッションではA,B2会場に おいて、「表面・界面」ならびに「無機材料」に関する ロ頭講演が行われました。

その後 10 時 20 分からは, 令和4年度論文賞の受賞 講演が行われました。「LED 光音響イメージングによ る血液体外循環回路内血液凝固予知保全のための輝度 範囲選定」のタイトルで,執筆者を代表して帝京科学 大生命環境学部の和部崇洋先生による講演が実施され ました。

その後,11時からは、令和4年度「小石眞純賞」受賞 者である 株式会社ジェイテクト 齊藤 利幸先生に よる受賞講演が「摩擦面生成物とトライボロジーに関 する研究」のタイトルで行われました。齊藤先生のこ れまでのご研究、そしてその成果がどのように製品開 発に結びついたかについて非常に興味深いお話をうか がうことができました。

昼食を挟んで13時からは、日本大学理工学部の浮谷 基彦先生による特別講演が「天然有機化合物を用いた 生物活性分子の合成と評価」のタイトルで実施され、 多くの注目を集めました。

その後,再び口頭講演が2会場に分けて実施された後, 15時10分からはポスター発表が3年ぶりに対面で行 われました。24件のポスター発表に対して,大変活 気のある質疑・応答が行われ,発表者の方,特にこれ まで対面で発表する機会が限られていた学生の皆さん にとっては,貴重な議論ができたとともに,学外の研 究者・学生と交流を深めるとても意義のある経験とな ったのではないかと思います。

ポスターセッション終了後,討論会は閉会となりまし たが,多くの参加者の方から,対面での学会参加は, 技術,また人の交流のためにも重要だとのご意見をい ただきました。これから Covid-19 の流行が終焉した後 でもオンラインでの討論の場は継続すると思いますが, やはり対面での議論の実り多さを実感する機会となり ました。 本討論会の開催は、非常に多くの方々のご尽力によ り可能となりました。リモート開催では従来以上に重 要となる討論会 HP の管理や要旨集のアップロードは 長谷川裕之副実行委員長と事務局のご尽力により円滑 に行われました。また、討論会の当日運営については、 酒井健一先生をはじめとする討論会委員を中心に円滑 に行われ、山内仁史専務理事には財政面でのアドバイ スを多くいただきました。また、多くの先生方には、 座長や賞の審査などでご協力いただき心よりお礼申し 上げます。

さらに、下記の方々から、討論会運営に協賛金、ご 寄付の形で多大なご支援をいただきました。心よりお 礼申し上げます。(敬称略,50音順)

エムディジャパン株式会社 株式会社 L.V.M.C. HTL 株式会社 小石眞純(材料技術研究協会 名誉会長) 小浦節子(材料技術研究協会 会長) 材料技術研究協会 表面改質研究会 株式会社 ジェイテクト 株式会社細川洋行 ユニ・チャーム株式会社

ロ頭講演賞・ポスター賞について

ロ頭講演・ポスター発表について,複数審査員によ る審査を実施し,特に優れた講演について,優秀ロ頭 講演賞,ゴールドポスター賞,シルバーポスター賞を 選考いたしました。以下に優秀ロ頭講演賞,ゴールド ポスター賞の受賞者(発表者)を記載させていただき ます。

優秀口頭講演賞受賞者(敬称略)

優秀口頭講演賞(10件)

1A-01 ホウ素・窒素共ドープダイヤモンド電極による
 低過電圧での高効率な CO₂電解還元(東理大理工)三
 宅祐大

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1B-03 微細藻類由来β - 1,3-グルカンの高分散化ナノ
 ファイバーの作製と水系塗料添加剤としての機能性
 (金沢工大院)小泉樹奈

1A-08 界面活性剤・高分子混合系の吸着挙動が電析 形状に与える影響(東理大理工)野口翔平

1A-09 天然由来触媒分子の固定化による固体表面の 不均一触媒機能の発現(東京高専)渡邉知樹

1B-08 光熱治療ナノ材料を指向した対称性崩壊アル ミニウムナノ材料の プラズモン波長制御と光熱変換 特性(日大院理工)矢葺大空

1B-11 光合成タンパク質を用いた Gabor フィルタ
 一素子による錯視検出(電気通信大学大学院)加古彰
 子

2A-01 リン脂質/グルコシルセラミド混合膜の物性に 及ぼす生理活性物質の添加効果(東理大理工)大長将 之

2B-03 La_{1-x}M_xNbO₄:0.02Dy³⁺ [M Gd,Y] ナノ結晶の 蛍光特性(近畿大院) 俵原南奈

2A-06 炭酸水を用いて調製したアルギン酸ゲルの物
 性評価と全層欠損創傷モデルマウスに対する創傷治癒
 効果の検証(東理大理)手島涼太
 2B-09 複合有機テンプレート法による多孔質球状シリ

カ粒子の調製 (千葉工大工) 菅野 透

ゴールドポスター賞 (8件)

(シルバーポスター賞はすべての発表者に授賞)
 P-01 キトサンナノファイバーフィルムの化学構造と
 表面構造が細胞増殖に与える影響(金工大院工バイオ・
 化学) 古谷幸一
 P-03 W/W'/O 分散系の逆相系界面重合法によるマイ
 クロカプセルの調製(新潟大学) 市原拓磨

P-07 フェロセン含有複合セラソームの形成と構造安 定性の制御(山形大院理工)鈴木皓大

P-12 CO₂ foam を反応場とした界面活性シリカナノ粒
 子の調製と物性評価(弘前大理工) 齊藤晴香
 P-13 六角板状酸化亜鉛粒子を用いた粒子担持薄膜の

作製(千葉工大工)曽我辺祐輝

P-14 分岐脂肪酸ナトリウム水溶液の泡沫特性・表面レ オロジーと pH の影響(東理大理工)山中雄太

P-16 微細球状バテライトとその構造色 (日大理工) 川島さゆり

P-20 粉末冶金法により作製した ODS フェライト鋼の 耐食性評価(久留米高専材料システム工)増永 光

編集後記

公開が遅れておりました Volume 40 の最終号(2022 年, Volume 40, No. 6) を公開いたします.お陰様で,現在も,会員の皆様から興味深い内容の 論文を御投稿いただいております。2023 年中には,公開の遅れを取り戻 し,会員の皆様にとって,より魅力のある媒体となるよう努力したいと 考えております(拙文は2022 年の最終号の編集後記ですが,2023 年の9 月に記しています).どうぞよろしくお願い申し上げます.

(浜松医科大学 三浦康弘)

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