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Enhancement of Surface Area of Cordierite Structure by Oxalic Acid Treatment for SCR Applications

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Abstract

Cordierite ($2Al_2O_3$-$5SiO_2$-2MgO) is the main carrier structure that enables the positioning of catalytic nanoparticles on the exhaust pipe in the selective catalytic reduction (SCR) applications. In order to be loaded more catalytic nanoparticles into this structure, it must be modified by acid treatment. In our study, these structures were treated with the different oxalic acid ratios for 2 and 4 hours. Brunauer, Emmet and Teller (BET) analysis were employed to measure the surface areas of acid-treated cordierite structures and scanning electron microscope-energy dispersive X-ray spectroscopy (SEM-EDS) analysis was used to examine morphological structures of them. In consideration of BET analysis result, the surface area measurements of these structures reached up to 163.601 m^2/g. The obtained value was about 327 times greater than the surface area of no treatment cordierite. In result of the SEM analysis, while the percentage of aluminum (Al) and magnesium (Mg) elements on the surface of these structures decreased, that of silicon (Si) increased. The reason of the high surface area is the formation of the pure amorphous silica due to the removal of Al and Mg ions from the surface of the cordierite. Thus, further coating of the catalytic nanoparticles on the cordierite surface could be allowed. As a result of this study, it could be said that the acid amount of the solution and the acid treatment duration enhances the surface area of the cordierite.

Keywords: Cordierite, Oxalic acid, Chemical etching process, BET, SEM

SCR Uygulamaları için Oksalik Asit İşlemi ile Kordiyerit Yapının Yüzey Alanının İyileştirilmesi

Öz

Kordiyerit ($2Al_2O_3$-$5SiO_2$-2MgO) seçici katalitik indirgeyici (SCR) uygulamalarında katalitik nanopartiküllerin egzoz borusu üzerinde konumlandırılmasını sağlayan ana taşıyıcı yapıdır. Bu yapıya daha fazla katalitik nanopartikül yüklenebilmesi için asit işlemi ile modifiye edilmesi gerekmektedir.

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Çalışmamızda bu yapılar 2 ve 4 saatlik sürelerde farklı oksalik asit oranları ile muamele edilmiştir. Asitle muamele edilmiş kordiyerit yapıların yüzey alanları Brunauer, Emmet ve Teller (BET) analizi ile ölçülmüş ve taramalı elektron mikroskobu-enerji dağılımlı X-ışını spektroskopisi (SEM-EDS) analizi ile morfolojik olarak incelenmiştir. BET analiz sonucuna göre bu yapıların yüzey alanı ölçümleri 163,601 m^2/g'a ulaşmıştır. Elde edilen bu değer, işlem görmemiş kordiyeritin yüzey alanından yaklaşık 327 kat daha fazladır. SEM analizi sonucunda bu yapıların yüzeyindeki alüminyum (Al) ve magnezyum (Mg) elementlerinin yüzdesi azalırken, silikonun (Si) yüzdesi artmıştır. Yüksek yüzey alanının nedeni, kordiyerit yüzeyinden Al ve Mg iyonlarının uzaklaştırılması nedeniyle saf amorf silikanın oluşmasıdır. Böylece, kordiyerit yüzeyi üzerinde katalitik nanoparçacıkların daha fazla kaplanmasına izin verilebilir. Bu çalışmanın sonucunda çözeltinin asit miktarının ve asitle muamele süresinin kordiyerit yüzey alanını arttırdığı söylenebilir.

Anahtar Kelimeler: Kordiyerit, Oksalik asit, Kimyasal aşındırma işlemi, BET, SEM

1. INTRODUCTION

SCR procedure is an effective emission control system aiming to reduce NO_x emissions from diesel engines. The main components of this procedure are reductant and catalyst. A variety of reductants such as ammonia (NH_3) [1,2], carbon monoxide (CO) [3], hydrogen (H_2) [4], and hydrocarbons (HC) [5,6] have been investigated for removing of NO_x emissions in the SCR systems.

Catalyst structures used in SCR applications consist of washcoat, active metal components, and substrate material. The basic function of the washcoat materials contributes to increase of surface area required for the dissipation and storing of catalytic metal components. Inorganic base metal oxides such as Al_2O_3 (alumina), SiO_2 (silica), TiO_2 (titanium oxide), CeO_2 (ceric oxide), ZrO_2 (zirconia), V_2O_5 (vanadium pentoxide), La_2O_3 (lanthanum oxide), ZnO (zinc oxide), and zeolites are used as the washcoat materials [7–12]. Some washcoat materials serve as secondary carrier support materials and others display catalytic activity. Active metal components display catalytic activity and consist of various precious metals (such as silver, copper, platinum, and palladium etc.), and oxides of base metals (such as vanadium, molybdenum, cerium, and tungsten etc.). Substrate materials are used as the main carrier structure in SCR applications. Cordierite ($2MgO \cdot 2Al_2O_3 \cdot 5SiO_2$) is preferred as a widespread carrier structure material, because of high heat resistance based on low thermal expansion property, adequate refractoriness property based on exceeding 1450°C of the melting point, pore size distribution and porosity which are facilitated both washcoat application and successful washcoat adhesion [9,13]. Monolith cordierite substrate is also widely used as the main carrier structure in other emission control systems such as diesel oxidation catalyst (DOC), lean NO_x trap (LNT), diesel particulate filter (DPF), and three way catalytic converter (TWC) applications [14–17].

In catalyst production, the cordierite coated with large amounts of the active catalytic metal components can be a significant parameter for its catalytic efficiency [18–20]. The increasing the surface areas of cordierite structures will allow more catalytic material to be coated on the surface [21]. The specific surface area of the cordierite is less than 1 m^2/g. Therefore, it is impossible to acquire highly dispersed and quite active catalysts by coating of the active catalyst components directly on the cordierite [22].

Researchers have conducted various studies on the modification of the cordierite. Table 1 summarizes the studies about increasing the surface area of the cordierite structure in the literature. All samples were immersed in different acid solutions at various durations. The surface areas of the cordierite increased from 0.5 m^2/g to 255 m^2/g in the study of Shigapov et. al. The surface area of the original cordierite was increased by 500 times due to acid treatment process [22].

Table 1. Summary of studies on the pretreatment of cordierite with various acids

Type of cpsi	Chemical etching conditions	BET area (m^2/g)	Ref.
400	No treatment (Blank cordierite)	0.5	[22]
400	10% Oxalic acid, boiling for 2h	38	
400	50% Oxalic acid, boiling for 2h	104	
400	50% Oxalic acid, boiling for 7h	255	
400	50% Oxalic acid, boiling for 1h	59	
400	8% EDTA, boiling for 1h	0.6	
400	50% Citric acid, boiling for 1h	11	
400	26% HNO_3, boiling for 2h	45	
400	1.25% HCl, at RT** for 18h	0.9	
400	37% HCl, at RT** for 24h	4.3	
400	10% HCl, at RT** for 24h and boiling for 1h	57	
400	37% HCl, boiling for 35 min.	28	
400	37% HCl, boiling for 2h	96	
400	37% HCl, boiling for 2h*	121	
400	37% HCl, boiling for 3h*	185	
400	37% HCl, boiling for 4h*	223	
400	37% HCl, boiling for 6h*	241	
400	37% HCl, boiling for 8h*	218	
300	20% HCl, boiling for 3h*	187	
300	20% HCl, boiling for 5h*	222	
400	No treatment (Blank cordierite)	0.5	[23]
400	20% H_2SO_4 at 90°C for 3h	81	
400	20% H_2SO_4 at 90°C for 6h	227	
400	No treatment (Blank cordierite)	0.17	[24]
400	65% HNO_3, boiling for 3h	116.88	
200	No treatment (Blank cordierite)	0.7	[25]
200	20% Oxalic acid for 6h	107.7	
200	35% Oxalic acid for 6h	148.2	
200	50% Oxalic acid for 6h	199.6	
400	No treatment (Blank cordierite)	1.25	[26]
400	68% HNO_3 at 110°C for 3h	7.88	
400	68% HNO_3 at 110°C for 6h	14.65	
400	68% HNO_3 at 110°C for 9h	23.69	
400	68% HNO_3 at 110°C for 12h	41.95	
200	No treatment (Blank cordierite)	0.7	[27]
200	50% Oxalic acid, boiling	200	
400	No treatment (Blank cordierite)	0.05	[28]
400	26% HNO_3, boiling for 2h	82.59	
400	No treatment (Blank cordierite)	0.5	[29]
400	50% Oxalic acid - 100°C for 3h	104.72	

*The solution was refreshed with new solution per every hour.

**RT is the abbreviation for room temperature.

In this study, it was aimed to enhance the cordierite surface area and to create a suitable surface for the positioning of catalyst nanoparticles before coating. The cordierite structure was pretreated by using different ratios of oxalic acid under various conditions. The surface areas of acid-treated cordierite structures were measured by BET analysis and morphologically examined by SEM-EDS analysis.

2. MATERIAL AND METHODS

2.1. Acid Treatment and Characterization

Ceramic cordierite having a cell density of 400 cpsi (cells per square inch) and a length of 100 mm, diameter of 130 mm was used in this study. Cordierite test specimens were cut from blank cordierite with the same dimensions as seen in Figure 1. These structures were pretreated at different durations and at various oxalic acid ($C_2H_2O_4 \cdot 2H_2O$) ratios in order to enhance the specific surface area at the first stage of catalyst production. The properties of oxalic acid are shown in Table 2. The conditions of experiments carried out with oxalic acid are summarized in Table 3.

Table 2. Oxalic acid specifications

Formula	= $C_2H_2O_4 \cdot 2H_2O$
Molar	= 126.07 g/mol
Assay (manganometric)	≥ 99.0%
Chloride (Cl)	≤ 0.002%
Sulfate (SO_4)	≤ 0.02%
Heavy metals (as Pb)	≤ 0.002%
Total nitrogen (N)	≤ 0.005%
Iron (Fe)	≤ 0.001%
Residue on ignition (800 °C as sulfate)	≤ 0.05%

Table 3. Conditions of oxalic acid treatment

Sample	Chemical etching conditions
S1	10% Oxalic Acid + 90% H_2O - 100°C for 2h
S2	10% Oxalic Acid + 90% H_2O - 100°C for 4h
S3	20% Oxalic Acid + %80 H_2O - 100°C for 2h
S4	20% Oxalic Acid + %80 H_2O - 100°C for 4h
S5	30% Oxalic Acid + 70% H_2O - 100°C for 2h
S6	30% Oxalic Acid + 70% H_2O - 100°C for 4h
S7	40% Oxalic Acid+ 60% H_2O - 100°C for 2h
S8	40% Oxalic Acid + 60% H_2O - 100°C for 4h
S9	50% Oxalic Acid + 50% H_2O - 100°C for 2h
S10	50% Oxalic Acid + 50% H_2O - 100°C for 4h

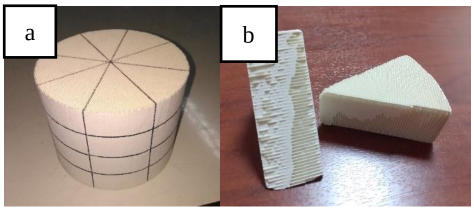


Figure 1. a) Determining appropriate dimensions, b) The cut cordierite structures

The cordierite structure was cut to appropriate dimensions for experiments as seen in Figure 1. All samples were treated with oxalic acid under specified conditions. Acid treatments were done by heater with the magnetic stirrer at 100°C as shown in Figure 2. After acid treatment, they were washed with pure water until the pH was neutral. The pretreated pieces were baked at 110°C for 1 hour to dry. Finally, they were then calcined at 550°C for 3 hours to remove impurities as shown in Figure 3.

Figure 2. Acid treatment of the samples

Figure 3. Calcination

After acid treatment, the surface areas of all samples were measured based on the nitrogen (N_2) gas adsorption technique at 77 K liquid nitrogen environment by BET method. The FEI Quante 650 FEG SEM was used to have information about the morphological structures of the samples. The samples coated with a conductive material (gold) of 2 Å/s were examined at an acceleration voltage of 20 kV. EDS detector was used to examine elemental analysis of the surfaces of the samples

3. RESULTS AND DISCUSSIONS

3.1. BET Surface Area Results

The BET analysis results of 10 samples were presented in Table 4. The cordierite structures consist of mostly SiO_2, MgO and Al_2O_3 structures. SiO_2 is acid, MgO is alkali and Al_2O_3 exhibits amphoteric. Therefore, when cordierite was treated with acid, the pure amorphous silica on the cordierite surface formed because of the removal of Mg ions and Al ions on the cordierite surface. This situation causes an increment in the cordierite surface area [21,22]. A surface area of 64.245 m^2/g was obtained for the S1 sample. When the amount of oxalic acid and the applied time were increased at the same temperature, the surface area value of the S10 sample was obtained as 163.601 m^2/g. This value is about 327 times higher than the surface area of the blank cordierite as seen from results in Table 4. In the literature, it was observed that increasing the duration applied at a constant acid rate increases the surface area of the cordierite [22,23,26]. Also, at a given duration in acid treatment process, the surface area can be increased as the acid content of the solution increases [22,25]. The duration applied and the change in acid content of the solution in this study are similar to the studies in the literature.

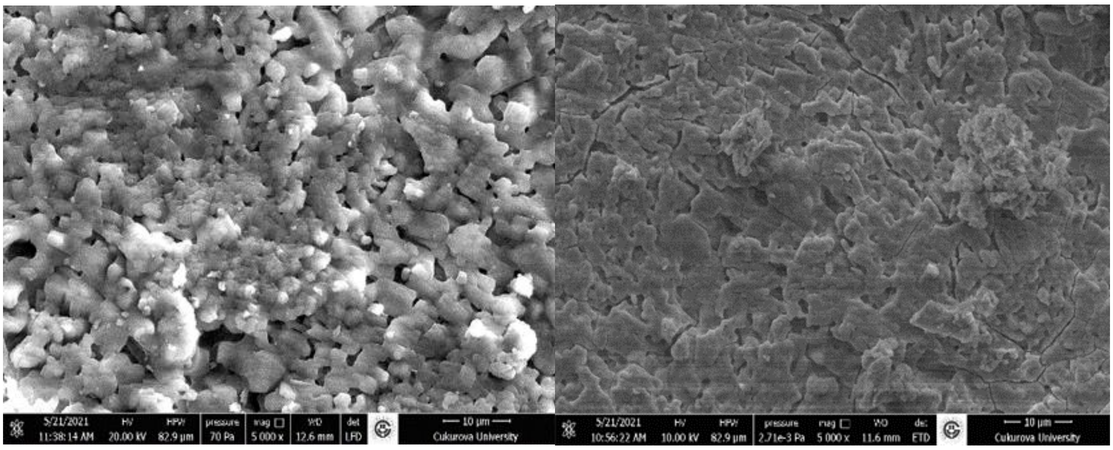
Table 4. BET analysis results of acid treated cordierite

Sample	Chemical etching conditions	BET surface area (m^2/g)
S1	10% Oxalic Acid + 90% H_2O - 100°C for 2h	64.245
S2	10% Oxalic Acid + 90% H_2O - 100°C for 4h	67.10
S3	20% Oxalic Acid + %80 H_2O - 100°C for 2h	70.536
S4	20% Oxalic Acid + %80 H_2O - 100°C for 4h	120.511
S5	30% Oxalic Acid + 70% H_2O - 100°C for 2h	71.061
S6	30% Oxalic Acid + 70% H_2O - 100°C for 4h	123.566
S7	40% Oxalic Acid + 60% H_2O - 100°C for 2h	74.633
S8	40% Oxalic Acid + 60% H_2O - 100°C for 4h	157.604
S9	50% Oxalic Acid + 50% H_2O - 100°C for 2h	85.235
S10	50% Oxalic Acid + 50% H_2O - 100°C for 4h	163.601

3.2. SEM-EDS Analysis Results

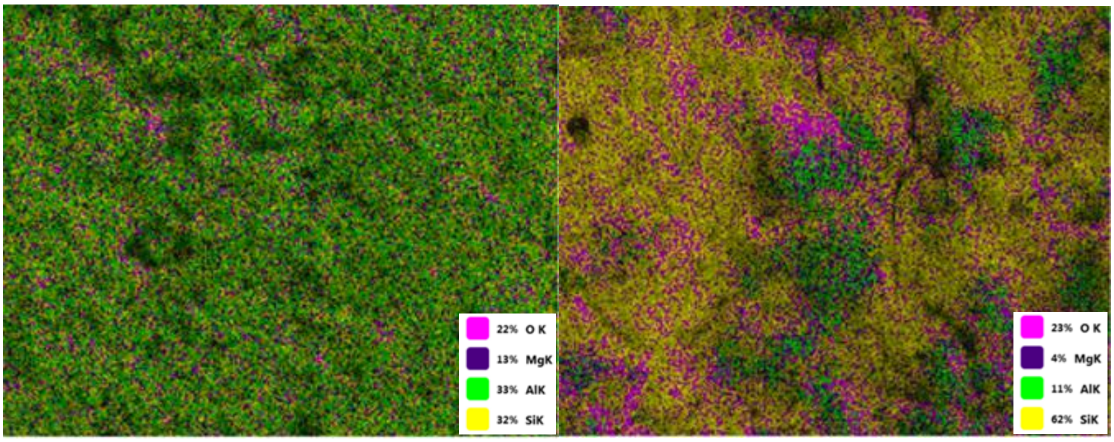
Surface morphologies of S10 sample and the cordierite were shown in Figure 4 by SEM images with 5000x magnification. When examined by SEM after chemical etching process, it can be discerned that cracks are formed on the surface of the cordierite structure on SEM image of S10 sample in Figure 4. It can be clearly demonstrated that these cracks, which are formed as a result of acid etching, lead to an increment in the surface area of cordierite. In this case, enhancement of the specific surface area of cordierite for catalyst production allows for the generation of more active sites and better distribution of active ingredients on the surface.

In Figure 5, SEM mapping images of acid etched cordierite and untreated cordierite structure are available. It can be seen that correspond to silicon (Si) as the yellow spots, aluminum (Al) as the green spots, magnesium (Mg) as the purple spots, and oxygen (O) particles as the pink spots on the SEM images. From the results of this analysis, it could be understood that after acid etching, Al and Mg ions are dissolved from the surface of the cordierite and their ratios decrease. Also, it caused the formation of amorphous silica. SEM-EDS results of acid etched cordierite and untreated cordierite structure are given in Figure 6. SEM-EDS results gave information about the change of cordierite surface after chemical etching. It can be discernible that the weight and atomic ratios of the elements vary. Finally, similar results were obtained for SEM-EDS and mapping analysis.



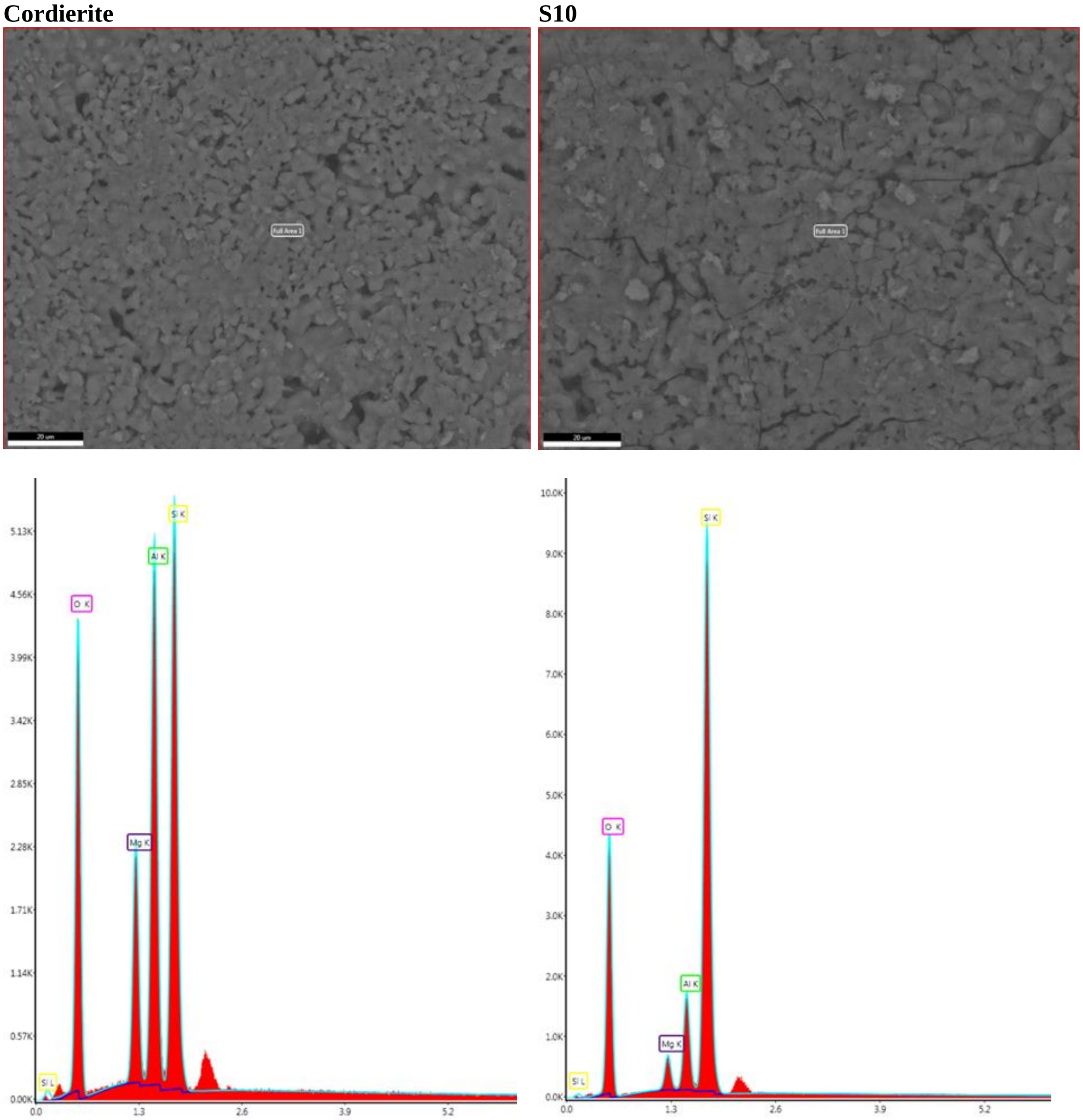
(a) **(b)**

Figure 4. SEM images of (a) Cordierite, (b) S10



(c) **(d)**

Figure 5. SEM mapping results of (c) Cordierite, (d) S10



Cordierite

S10

Elements	EDS		
	Weight (%)	**Atomic (%)**	**Net int.**
O K	42.77	55.78	679.75
Mg K	9.17	7.87	400.9
Al K	21.16	16.36	934.25
Si K	26.91	19.99	1043.58

Elements	EDS		
	Weight (%)	**Atomic (%)**	**Net int.**
O K	47.92	61.44	687.53
Mg K	2.69	2.27	101.66
Al K	6.98	5.31	303.08
Si K	42.42	30.98	1848.69

Figure 6. SEM-EDS images and results

4. CONCLUSION

In this study, it could be concluded that increasing the duration applied at a constant acid rate increases the surface area of the cordierite. Also, at a given duration in chemical etching process, the surface area can be enhanced as the acid amount of the solution rises. The surface area value of 163.601 m^2/g obtained by acid treatment of cordierite increased approximately 327 times from the value before acid treatment. As can be seen from the SEM analysis results, the percentage of Mg and Al ions decreased and the percentage of Si increased. After the acid treatment of cordierite, the pure amorphous silica was formed due to the removal of Mg and Al ions on the cordierite surface. Thus, the SEM analysis results supported the change of the cordierite surface in relation to the increase in the BET surface area values of the cordierite structure. It was understood that after acid treatment, the rough and porosity surface structure of cordierite is preserved. Increasing the specific surface area of cordierite for catalyst production can be resulted in better distribution of active ingredients on the surface and formation of more active sites.

5. INFORMATION

This study belongs to the PhD graduation project.

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