

Article

# Formation Mechanism of MgO Containing Inclusions in the Molten Steel Refined in MgO Refractory Crucibles

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**Abstract:** In order to study the influence of MgO refractory on inclusions during the steelmaking process, laboratory quasi-equilibrium experiments were performed in MgO crucibles at 1873 K. It was found that there were no MgO-containing inclusions formed when ultra-low Al ultra-low C steel was used. When the contents of dissolved Al and C in steel changed, different composition ranges led to different types of inclusions. It was shown as well that for both low Al high C steel and high Al low C steel, the order of average MgO content in inclusions from large to small was: MgO-10% slag addition > MgO-free slag addition > no slag addition. Based on the results, a simple formation mechanism of MgO-containing inclusions in the steel with different compositions in MgO crucibles was proposed. The results could provide guidance for the reduction of MgO-containing inclusions in the industrial production of steels.

**Keywords:** non-metallic inclusions; MgO crucible; MgO reduction; steel composition; slag

## 1. Introduction

Non-metallic inclusions are originated from the deoxidation of molten steel [1]. Most of the inclusions would be removed by Stokes floatation, bubble floatation [2,3], refractory adhesion or slag absorption. The remained inclusions in steel are usually detrimental to steel properties depend on the quantity, size and composition of inclusions [4,5]. One of the most important ways to decrease the detrimental effect is reducing the inclusion size and controlling the inclusion composition [4].

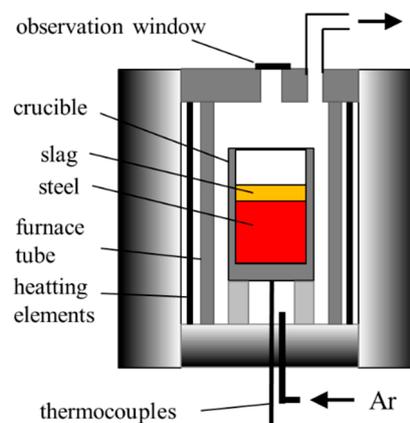
MgO-based refractories, such as MgO-C, MgO-CaO, MgO-Al<sub>2</sub>O<sub>3</sub> and MgO-Cr<sub>2</sub>O<sub>3</sub> types, are widely used as glaze materials of ladle and tundish, which results in the inevitable generation of MgO-containing inclusions in steels. MgO-containing inclusions usually have small size but high melting temperature and hardness and would further affect the cleanliness and property of steels. Thus, it is important to keep an eye on the formation behavior of MgO-containing inclusions in the steel melted in MgO-based refractories. There have been many studies in the regard. Li et al. [6] studied the reactions between MgO-C refractory, molten slag and metal using a high temperature X-ray radiographic apparatus. It was found that the local corrosion was regarded as due to the cyclic dissolution of MgO and graphite in the refractory into slag and metal phase. Harada [7] and Huang et al. [8] studied the dissolution behavior of Mg from MgO refractory into Al-killed steels by experiment and modeling, respectively. The reaction between slag and refractory also has a relevant influence on the formation of inclusions. The corrosion of MgO-based refractories by slag in steelmaking process has been investigated [9–12], it was found that a layer of MgO and spinel would

be formed at the slag/refractory interface [13]. On an industrial scale, Du [14] found that the ladle glaze was a major source of inclusions in the ladle. Lehmann [15] found that the reaction between a MgO-based tundish refractory and an Al-killed steel would induce a pollution of the liquid steel based on an experimental study. The formation of inclusions in Al-killed molten steel in MgO-C crucibles was investigated in laboratory as well [16,17]. It was concluded that the reaction between MgO and C occurred in the MgO-C refractory. The oxide inclusions formed in the steel were shown mainly to consist of MgO, Al<sub>2</sub>O<sub>3</sub> or mixture of them [16,17]. The current authors also found that the MgO content in inclusions increased with increasing the holding time after the deoxidation of molten steel by aluminum in a MgO crucible [18]. In recent years, Liu investigated the effect of refining slag [19,20] and magnesia-carbon [21] or magnesia-chromite [22] refractories on the inclusion formation and transformation in Al-killed steels. The dissolution behavior of Mg from the refractory into the steel and the effect of the refractory on the kinetics of the generation and transformation of inclusions were discussed.

In previous studies, MgO-C refractory or crucibles were mainly used; the main focus was on the reaction mechanism at the interface of refractory-slag or refractory-steel. However, it is difficult to distinguish whether MgO is reduced by carbon in refractories or steel or by aluminum in steel. Moreover, it needs to be clarified that the reduced MgO is from refractory or slag. Thus, in the current work, laboratory experiments were performed to investigate the effects of carbon and aluminum contents in steel and MgO content in slag on the formation of MgO-containing inclusions in molten steel refined in MgO crucibles.

## 2. Experimental Procedure

Quasi-equilibrium experiments were carried out in a silicon-molybdenum resistance furnace to investigate the effect of compositions of steel and slag on the formation of inclusions in MgO refractories. The experimental setup is illustrated in Figure 1 [18]. Approximately 100 g steel and 50 g slag (in heats with slag addition) were melted in a MgO crucible under purified argon atmosphere at 101 kPa and 1873 K. After holding for two hours—when the reaction was estimated to reach equilibrium—the steel sample was quenched by water. Several groups of experiments with different contents of Al and C in steel and different slags were performed, as listed in Table 1, including the group of ultra-low aluminum ultra-low carbon (ULAULC), the group of low aluminum high carbon (LAHC), the group of ultra-low aluminum high carbon (ULAHC) and the group of high aluminum low carbon (HALC). The steels used for the experiments were prepared by adding required elements into the melted electrolytic iron (EI), whose composition is listed in Table 2, in a vacuum induction furnace. The initial compositions of the two kinds of slag are listed in Table 3, the basicity ( $R = \text{CaO}/\text{SiO}_2$ ) and  $\text{CaO}/\text{Al}_2\text{O}_3$  ratio of the two slags are the same, the difference is that Slag A is MgO free while Slag B contains 10 wt. % MgO.



**Figure 1.** Schematic of experimental setup (Adapted from [18], with permission from John Wiley and Sons, 2020.).

**Table 1.** Steel and slag information of experiments.

Group	Heat no.	Slag (R = 7, 50 g)	Steel Preparation (around 100 g)
ULAULC	1	No slag	Electrolytic iron (EI)
	2	Slag A	
	3	Slag B	
LAHC	1	No slag	1 kg EI + 10 g C melted in Al <sub>2</sub> O <sub>3</sub> crucible using VIF
	2	Slag A	
	3	Slag B	
ULAHC	1	No slag	1 kg EI melted in graphite crucible using VIF
	2	Slag A	
	3	Slag B	
HALC	1	No slag	1 kg EI + 10 g Al melted in Al <sub>2</sub> O <sub>3</sub> crucible using VIF
	2	Slag A	
	3	Slag B	

Note: ULAULC means ultra-low aluminum ultra-low carbon, LAHC means low aluminum high carbon, ULAHC means ultra-low aluminum high carbon, HALC means high aluminum low carbon, VIF means vacuum induction furnace.

**Table 2.** Composition of electrolytic iron (wt. %).

Element	C	Si	Mn	P	S	T.Al	T.Mg	T.O
Content	0.0024	<0.0005	0.0001	0.0004	0.0006	0.0018	<0.0004	0.0105

Note: T.Al is total aluminum, T.Mg is total magnesium and T.O is total oxygen.

**Table 3.** Initial component of slag before experiments (wt. %).

Slag	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO
A	48.1	6.9	45.0	0.0
B	43.2	6.2	40.6	10.0

After experiments, the contents of carbon and total oxygen (T.O) of steel samples were analyzed using infrared analysis, the contents of aluminum and magnesium were analyzed using ICP-AES (Thermo Fisher Scientific Inc., Massachusetts, MA, United States). The composition of slags was detected by X-ray fluorescence analysis. Inclusions in the cross-section of each sample were detected using Aspex, an automatic scanning electrical microscopy, the scanned area of each sample was approximately 10 mm × 10 mm and inclusions larger than 0.5 μm were detected.

### 3. Variation of the Composition of Steel and Slag

The compositions of steel and slag before and after quasi-equilibrium experiments are presented in Table 4. In the group of ULAULC, the content of total magnesium (T.Mg) in steel showed small variations and kept trace amount after experiments no matter the slag was added or not. In the group of LAHC, however, because the steel was prepared by melting electrolytic iron and graphite in a corundum crucible using vacuum induction furnace, some amount of dissolved aluminum ([Al]) were presented in the steel. In the cases of slag-free and slag B (10% MgO contained) addition, the contents of [Al] and T.Mg increased after experiment, while in the case of slag A (MgO-free) addition the contents showed limited variations. By contrast, in the group of ULAHC, the steel was prepared by melting electrolytic iron in a graphite crucible so as to avoid the generation of [Al] (<10 ppm in the current work). After experiments, the contents of [Al] and T.Mg slightly increased while the C content decreased from saturation to approximately 3.8%. In the group of HALC, when slag was not added, the content of [Al] in the steel decreased slightly after experiments, but in the case of slag addition the content of [Al] decreased obviously, which should be owing to the reduction of SiO<sub>2</sub> in the slag, the decrease of SiO<sub>2</sub> in the slag shown in the table could be an evidence. The content of T.Mg in the HALC

group increased more or less after each quasi-equilibrium experiment. The change of steel composition mentioned above would lead to different formation characteristics of inclusions in steel.

**Table 4.** Steel and slag composition before and after quasi-equilibrium experiments (%).

Group	No.	Slag	Steel Composition				Slag Composition after Experiment				
			[Al]	T.Mg	C	T.O	CaO	Al <sub>2</sub> O <sub>3</sub>	MgO	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>
ULAULC	0		0.0008	<0.0004	0.0024	0.0105					
	1	none	<0.0005	<0.0005	-	0.0060					
	2	A	<0.0005	<0.0005	-	0.0041	48.55	32.20	9.04	6.69	3.01
	3	B	<0.0005	0.0005	-	0.0081	48.19	31.45	9.43	6.20	4.35
LAHC	0		0.0021	<0.0004	0.84	-					
	1	none	0.0032	0.0068	-	0.0160					
	2	A	0.0016	0.0003	0.78	0.0123	50.45	33.40	7.46	6.64	1.62
	3	B	0.0075	0.0039	-	0.0106	47.97	33.60	9.91	6.33	1.73
ULAHC	0		0.0006	<0.0004	4.37	0.0181					
	2	A	0.0012	0.0004	3.77	0.0167	50.61	33.78	9.09	6.52	1.86
	3	B	0.0012	0.0007	3.85	0.0133	50.52	33.67	9.54	6.27	
HALC	0		0.40	<0.0004	0.0062	-					
	1	none	0.32	0.0010	-	0.00050					
	2	A	0.0096	<0.0005	-	0.00044	50.31	33.26	8.16	5.61	1.73
	3	B	0.0110	0.0006	-	0.00043	48.52	34.49	9.81	5.14	1.58

Note: No. 0 in each group means before experiment, “-” means “No detection”.

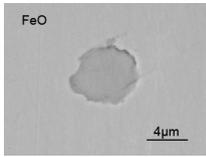
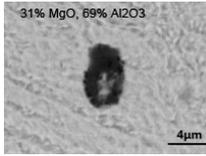
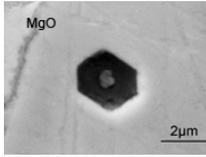
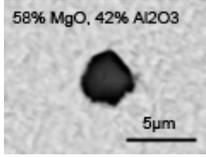
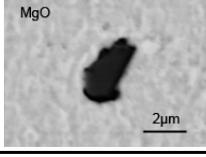
From the slag results, although it was MgO-free in Slag A, after experiment the MgO content in the slag increased significantly, which was owing to the erosion of the MgO crucible by slag. The SiO<sub>2</sub> content in slag showed not significant variation in all groups except the group of HALC, indicating that the SiO<sub>2</sub> in slag could only be significantly reduced under the condition of high [Al] content in steel. Besides, the Al<sub>2</sub>O<sub>3</sub> content in slag after experiments decreased in all groups, which might be due to the reaction between the Al<sub>2</sub>O<sub>3</sub> in slag and the MgO in crucible at the slag-crucible interface. However, the exact mechanism was still unclear. The higher content of iron oxide in the slag of group ULAULC was mainly resulted from the higher dissolved oxygen in the steel.

#### 4. Characteristics of Inclusions

The type and typical morphology of inclusions after each experiment are summarized in Table 5. In the group of ULAULC, there was no other type inclusions other than FeO was detected. It was indicated that the crucible refractory would not be peeled off easily by the erosion of still molten steel, estimating that the generation of MgO-containing inclusions in other groups of experiments should be mainly caused by chemical reactions.

In all experiments of LAHC group, the inclusions were mainly MgO-Al<sub>2</sub>O<sub>3</sub> spinel type, some of which were rich in MgO. By contrast, almost all inclusions in the group of ULAHC were pure MgO. The difference could be attributed to the difference of [Al] content in steel. It shows that only tens of ppm of [Al] in high carbon steel could lead to the formation of spinel in MgO crucibles. In the group of HALC, in the cases of slag-free and Slag A addition, inclusions were mainly MgO and MgO-rich spinel. However, when Slag B was added, the inclusions in the steel were almost pure MgO. Figure 2 shows the element mapping of typical MgO-rich spinel inclusions in the group of HALC, indicating that the inclusions were composed of a spinel core and a pure MgO surrounding-layer.

Table 5. Inclusion characteristics after quasi-equilibrium experiments.

Group	No.	Slag	Inclusion	
			Morphology	Type
ULAULC	1	none		FeO
	2	A		FeO
	3	B		FeO
LAHC	1	none		spinel
	2	A		spinel
	3	B		spinel
ULAHC	2	A		MgO
	3	B		MgO
HALC	1	none		MgO + spinel
	2	A		MgO + spinel
	3	B		MgO

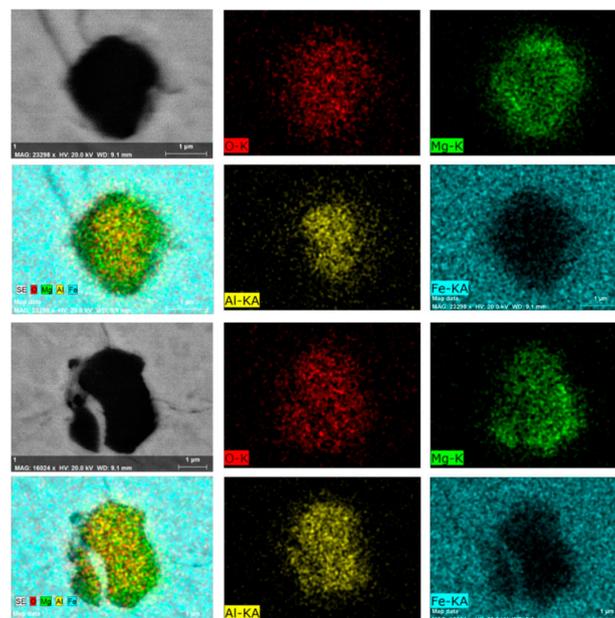


Figure 2. Element mapping of MgO-rich spinel inclusions in the group of HALC.

Although the inclusion types looked like the same both in the group of LAHC and in two experiments of group HALC, in fact, the average MgO content in the inclusions was different. The statistical results were analyzed using Aspex, as shown in Figure 3. It was indicated that for both the group of LAHC and the group of HALC, the order of MgO content from large to small was slag

B > slag A > slag-free. When slag was not added, the MgO in inclusions could only come from the reaction between steel and crucible. Since the crucible was solid and dense, the mass transfer in the crucible was very slow. On the other hand, spinel—the reaction product at the steel-crucible interface—would hinder the diffusion of elements and further reduce the reaction rate. When slag was added, MgO in inclusions could be partially generated by the reaction between molten steel and liquid slag, in which the mass transfer was faster. In addition, due to the better wettability between slag and crucible, MgO crucibles would be more easily eroded by slag, leading to the saturation trend of MgO in slag. Moreover, the addition of slag could make the Al<sub>2</sub>O<sub>3</sub> inclusions in steel be removed more, thus reducing the Al<sub>2</sub>O<sub>3</sub> content in inclusions after experiments. Under the addition of 10% MgO slag, MgO was saturated in the slag from the beginning of experiment, thus resulting in higher MgO activity in the slag and more MgO content in inclusions.

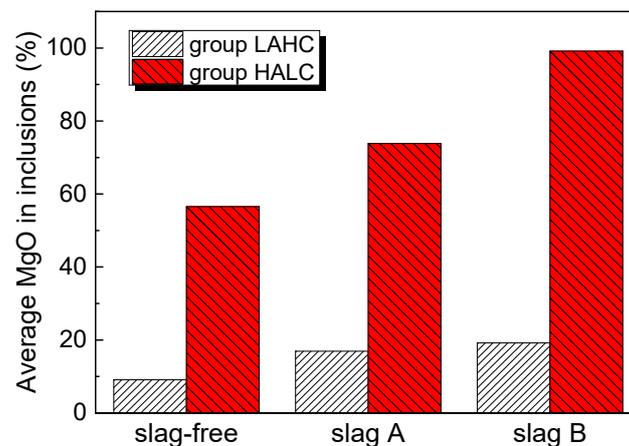
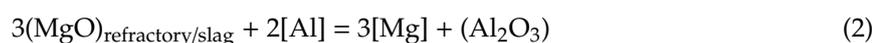
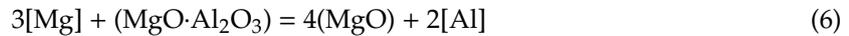


Figure 3. Average MgO content in inclusions after quasi-equilibrium experiments.

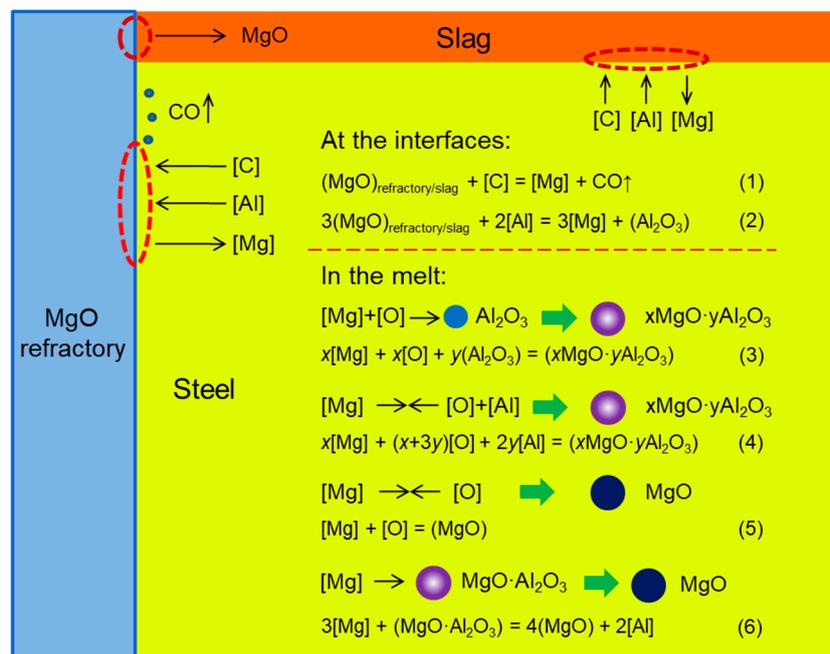
## 5. Formation Mechanism of MgO-Containing Inclusions

According to the experimental results, a simple formation mechanism of MgO-containing inclusions in the steel with different compositions in MgO crucible is proposed, as illustrated in Figure 4. In ultra-low Al ultra-low C steel ([Al] < 0.0010%, C < 0.0025%, corresponding to the group of ULAULC), the molten steel and the refractory are substantially unreactive. In high C steel (C > 0.5%, corresponding to the groups of LAHC and ULAHC), at the interface of crucible-steel or slag-steel, Reaction (1) will take place, that MgO will be reduced by [C] to generate [Mg] and CO gas, which is consistent with the phenomenon that many gas holes existed on the surface of steel samples of group LAHC and ULAHC. When a certain amount of [Al] is contained in the steel (approximately > 0.0020%, corresponding to the groups of LAHC and HALC in the current work), Reaction (2) may also happen, that MgO will be reduced by [Al]. When the generated [Mg] diffuses into the melt, it will react with the residual Al<sub>2</sub>O<sub>3</sub> or [Al] to form MgO-Al<sub>2</sub>O<sub>3</sub> type spinel in the LAHC and HALC steels, shown as Reaction (3) or (4). In the high C steel with ultra-low Al content ([Al] < 0.0010%, corresponding to the group of ULAHC), [Mg] and [O] will react with each other directly to generate pure MgO in the melt, shown as Reaction (5). In the high Al low C steel ([Al] > 0.01%, C < 0.006%, corresponding to the group of HALC), [Mg] is mainly generated by the reduction of MgO in slag or refractory by [Al], shown as Reaction (2). The inclusions in the group of HALC were consisted of a spinel core and a pure MgO surrounding layer. Accordingly, inside the high Al low C molten steel, Reaction (3) or Reaction (4) occurs first, then, when the MgO in spinel reaches saturation, pure MgO will be formed on the surface of spinel according to Reaction (5) or Reaction (6).





Based on the aforementioned analysis, MgO-containing inclusions are originated from the reduction of MgO in refractory or slag by carbon in the situation of high carbon steel or by aluminum when the Al content in steel reaches tens of ppm. Thus, in order to reduce the formation of MgO-containing inclusions in the industrial production of steels, first of all, the activity of MgO in the refractory and slag should be decreased. Considering that the slag with unsaturated MgO content will intensify the erosion of refractories, it is more feasible to reduce the activity of MgO in refractories, which can be realized by replacing the MgO refractory with other MgO-based refractories, such as MgO-Al<sub>2</sub>O<sub>3</sub> and MgO-CaO types, especially for Al-killed steels and high carbon steels. It should be pointed out that MgO-Al<sub>2</sub>O<sub>3</sub> type refractories are not suitable for high carbon steels with strict restrictions on Al<sub>2</sub>O<sub>3</sub> content in inclusions. Meanwhile, the supersaturation of MgO in slag should also be avoided. Moreover, MgO-C refractories, which are widely used in ladle slag line, have poor wettability with slag, but good wettability with molten steel, so they are easy to react with molten steel, resulting in the amount increase of MgO-containing inclusions. Therefore, it is necessary to minimize the weight fluctuation of molten steel and slag in each heat during the production process so as to keep the slag level stable during the refining process and reduce the reaction between the molten steel and MgO-C refractories.



Ultra-low Al ultra-low C steel: steel and the refractory are substantially unreactive;  
 Low Al high C steel: interface reactions (1) and (2), melt reaction (3) and (4);  
 Ultra-low Al high C steel: interface reaction (1), melt reaction (5);  
 High Al low C steel: interface reaction (2), melt reactions (3) or (4) first, then (5) or (6).

Figure 4. Illustration of inclusion formation in molten steel in MgO crucibles.

## 6. Conclusions

Quasi-equilibrium experiments were performed in the laboratory to investigate the effect of compositions of steel and slag on the formation behavior of MgO-containing inclusions in the molten steel refined in MgO crucibles. Some conclusions were obtained as follows:

- (1) In ultra-low Al ultra-low C steel ( $[Al] < 0.0010\%$ ,  $C < 0.0025\%$ ), there was no MgO-containing inclusions other than FeO formed.
- (2) In ultra-low Al high C steel ( $[Al] < 0.0010\%$ ,  $C > 0.5\%$ ), almost all inclusions were pure MgO. When the  $[Al]$  content in steel increased to larger than  $0.0015\%$ , inclusions were changed to spinel, some of which were rich in MgO.
- (3) In high Al low C steel ( $[Al] > 0.01\%$ ,  $C < 0.006\%$ )—in the cases of slag-free and addition of MgO-free slag—inclusions were mainly MgO and MgO-rich spinel, while with the addition of 10% MgO contained slag, inclusions were almost pure MgO.
- (4) For both the low Al high C steel and the high Al low C steel, the order of MgO content from large to small was: slag B > slag A > slag-free. Based on the experimental results, a simple formation mechanism of MgO-containing inclusions in the steel with different compositions in MgO crucibles was proposed.

**Author Contributions:** W.Y. and L.Z. conceived and designed the experiments; Y.L. performed the experiments; Y.L. and W.Y. analyzed the data; Y.L. and W.Y. wrote the paper. All authors have read and agreed to the published version of the manuscript.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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