

Cooperative Research on Standardization of Test Method for Exfoliation
Corrosion Susceptibility in Al-Li Series Alloys (1st Report)

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ABSTRACT In order to standardize test method for relative exfoliation corrosion susceptibility in Al-Li series alloys, cooperative research has been planned and experimental programs for accumulating basic corrosion data were conducted. The materials tested were Al-Li series alloy sheets of 2090, 2091 and 8090 in T3 and T8 conditions and conventional alloy sheets of 2024-T351 and 7075-T651 as reference. Accelerated laboratory corrosion tests were performed, and these were EXCO (ASTM G34-90), modified EXCO, ASSET (ASTM G66-86) and MASTMAASIS (ASTM G85-85). Effect of Li-loss layer in the surface region, suspending direction of specimens, solution temperature and period of exposure were also examined. The differences in corrosion behaviors among these four test methods were revealed, and thus it was suggested that EXCO test would be useful for evaluating the relative exfoliation corrosion susceptibility in Al-Li series alloys.

*Keywords : aluminum-lithium alloys, exfoliation corrosion, pitting corrosion,
accelerated test, laboratory test*

1. INTRODUCTION

Aluminum-Lithium alloys are lighter, and have the higher value of elastic modulus than conventional aluminum alloys. Thus, it is expected as next generation aerospace material. However, there were very few corrosion data, and the results were fluctuated. That may be caused by such following items as accelerating corrosion condition, specimen condition, evaluation method etc., are not established. An effect of Li-loss layer in the surface region on corrosion behavior is a peculiar problem for Al-Li series alloys.

EXCO test per ASTM G 34, ASSET test per ASTM G 66, and MASTMAASIS test per

ASTM G85 have been used for evaluating susceptibility to the exfoliation corrosion for conventional aluminum alloys, but, it has been doubtful whether these tests have been useful for Al-Li series alloys. Thus, it needs to standardize test method for relative exfoliation corrosion susceptibility in Al-Li series alloys.

In this paper, in order to standardize test method, some accelerated tests relating to exfoliation corrosion were performed for some kind of Al-Li series alloys and conventional alloy 2024-T351 and 7075-T651 as reference. Effect on the existence of Li-loss layer in the surface region, suspending direction of specimen, solution temperature and period of exposure were examined. After immersion into the test solution, appearance and cross-section microstructure of specimens were evaluated for each alloy and the relative degree of susceptibility for the exfoliation corrosion was determined. The test results were summarized and discussed.

2. EXPERIMENTAL PROCEDURE

2.1 Specimen

Tests were accomplished to discuss the effect of specimen conditions on the test results, such as kinds of alloy and temper, existence of Li-loss layer in the surface region and size of specimen.

Al-Li series alloy 2090, 2091, 8090 sheets in T3 and T8 conditions and conventional alloy sheets of 2024-T351 and 7075-T651 as reference were used for the tests. The specimen size was 50mm (longitudinal direction) \times 100mm per ASTM G 34. To effect of the Li-loss layer in the surface region, as received and removed specimens were tested.

Before testing, the specimens were prepared as follows; (1)degreasing by organic solvent, (2)alkaline cleaning (immersion in 10% NaOH solution at 40°C for 1min., (3)neutralization (immersion in 10% HNO₃ solution at R.T. for 1min.) and (4)drying. After that preparation, the specimens were kept in descicator.

2.2. Experimental Method

2.2.1 Accelerated laboratory corrosion method

In order to study the suitable test condition, the following four candidate test methods about the exfoliation corrosion were tried ; (1) EXCO (ASTM G34-90), (2) modified EXCO, (3) ASSET (ASTM G66-86) and (4) MASTMAASIS (ASTM G85-85). Modified EXCO test was planned to moderate the corrosion severity. Nitric acid concentration of the solution was reduced to half that of the EXCO test solution.

And, in order to settle the most suitable conditions for Al-Li series alloys, the details of test conditions were controlled as follows.

(1) EXCO test

Test was performed under the condition as specified in ASTM G34 and some tests were parametrically deviated from standard method. Parameters for test conditions are as follows (condition underlined is standard one.) ;

- Li-loss layer ; Not removed (as received), Removed
(0.5mm thick surface layer was removed by emery papers for the latter specimens.)
- Supporting of specimen ; Vertical, Horizontal
- Period of exposure ; 5, 12, 24, 48, 72 and 96 hours
- Temperature of solution ; 25, 35°C

(2) Modified EXCO test

The test parameters are the same as those of EXCO test. Period of exposure (48, 96

and 168 hours).

(3) ASSET test

The test parameters other than period of exposure are same as EXCO test. Test period of exposure was 12, 24 and 48 hours (standard period of exposure is 24 hours).

(4) MASTMAASIS test

Basic test was performed under the condition as specified in ASTM G85 and some tests were parametrically deviated from standard test. Parameters for test conditions are as follows (condition underlined is standard one.) ;

- Li-loss layer ; Not removed (as received), Removed
- Time of exposure ; 1 week, 2 weeks
- Temperature of solution ; 25, 35°C

2.2.2 Evaluation method of Result

Specimens were cleaned using the running water, as soon as the exposure in the test solution was finished. Then, rating of the appearance was performed before the surface of specimen dried. The cross-section microstructure ($\times 100$) of each specimen was also examined. The cross-section of specimen cut parallel to the grain direction was microscopically observed.

The degree of susceptibility for the exfoliation corrosion was determined from the appearance and cross-section microstructure. The following codes, classification and level (4 grades of corrosion) were used for rating. The rating code of exfoliation corrosion showed by ASTM G 34 was adopted as reference.

N: None (Not corroded)

EA~ED: Exfoliation Corrosion (EA; Light, ED; heavy)

PA~PD: Pitting Corrosion (PA; Light, PD; heavy)

For some case, only corrosion type was decided, and the code was denoted by "E" or "P".

3. RESULT AND DISCUSSION

Many test data were accumulated. As a result, it was supported that EXCO test would be suitable, because exfoliation corrosion was generated mainly rather than pitting corrosion and it was relatively easy to classify with ratings.

Thus, the results of EXCO test is described in detail below. As for modified EXCO, ASSET and MASTMAASIS tests, the results are described briefly.

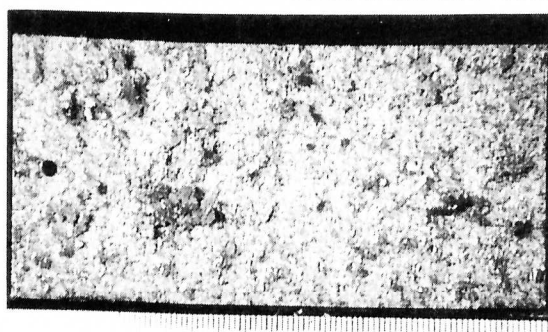
3.1. EXCO Test

The summary of the results of EXCO test is listed in Table 1. And, Fig.1 shows the example of appearance and cross-section microstructure of the 2091-T8 tested under the standard condition. The exfoliation corrosion is found to occur in this photograph. The surface of the specimen is covered by corrosion products colored red or light brown, and the surface layer is peeled away severely.

Judging from Table 1, the type of corrosion attack of Al-Li series alloys for this EXCO test is mainly exfoliation corrosion. And then the difference in corrosion resistance between T3 and T8 temper for Al-Li series alloys is easily distinguished. T3 temper alloys exhibit better exfoliation corrosion resistance than T8 temper alloys. For example, the rating code of 2090-T3 under the standard condition was "PA", while that of 2090-T8 was "EC". Corrosion resistance of T8 temper Al-Li series alloys was intermediate between 7075-T651 and 2024-T351.

TABLE 1 SUMMARY OF RESULTS FOR EXCO TEST

Test condition			2090		2091		8090		2024	7075
			T3	T8	T3	T8	T3	T8	T351	T851
Standard (as specified)			PA EA	EC EB	PB EA	EC EB	PA/C EA	EB/A EB	PB EA	EC EC
deviated	Li-loss layer removed		PC PC	EC EC	PB PC	EC EC	PB PC	EB EB	PC PC	ED ED
	Suspending horizontally		PA PB	EC EC	PC PB	EC EC	PB PC	EA EA	PB PB	ED ED
	Exposure time	5h	PA PA	PA PA	PA PA	PA PA	PA PA	PB PA	N N	PB PA
		12h	PB PA	EA EA	PB PA	PB EA	PB PA	EA PA	PA PA	EB EA
		24h	EA PA	EA EA	EA PB	EA EB	EA PB	EA EB	PB PB	EA EB
		72h	EA P/EA	ED EC	EA PB	ED EC	EA PB	ED EC	EA EA	ED ED
		96h	EA PA	ED ED	EA PB	ED EC	EA PB	ED ED	EA EB	ED ED
		Solution temp. (35℃)		EA	EB	PA	EC	EA	EC	PA



(a) Appearance



(b) Cross-section Microstructure

FIG. 1 APPEARANCE AND CROSS-SECTION MICROSTRUCTURE OF 2091-T8 TESTED UNDER THE STANDARD CONDITION. (EXCO TEST)

Effect of exposure period (5, 12, 24, 48(standard), 72 and 96 hours) was examined. The degree of corrosion increased with the elapse of exposure period. All of Al-Li series alloy specimens exposed for 24 hours showed the rating code of "EA", and no difference among alloys was found. However, for the specimens exposed for 48 hours, there was a clear difference between tempers, namely rating code of T3 was "EA" and that of T8 was "EB". And, during the period from 72 to 96 hours, T8 temper alloys were attacked more heavily, leading to a change of code to "EC" or "ED", while T3 temper alloys remained "EA". Then, it appeared that the type and tendency of corrosion attack are not changed during exposure over 48 hours, and thus the corrosion performance is assessed well at 48 hour exposure.

As for the effect of Li-loss layer in the surface region, the specimen removed the layer exhibited a heavier corrosion attack relatively than that not removed.

The extent of the exfoliation corrosion was relatively more severe under the test temperature of 35°C, compared with that under the standard test temperature (25°C). The tendency in corrosion behavior at 35°C was nearly same as that at 25°C.

For the suspending direction (horizontally or vertically) of specimens, there was no difference in the test results in any direction.

From the above results, it is concluded that the EXCO test in accordance with ASTM G34 is suitable to assess the exfoliation corrosion susceptibility in Al-Li series alloys.

3.2 Modified EXCO Test

The summary of test results of modified EXCO test is listed in Table 2. Generally, The corrosion tendency in modified EXCO test was not severe compared with that in the EXCO test and pitting corrosion tended to occur.

TABLE 2 SUMMARY OF RESULTS FOR MODIFIED EXCO TEST

Test condition			2090		2091		8090		2024	7075
			T3	T8	T3	T8	T3	T8	T351	T651
Standard (as specified)			N EA	N/EA EB	P PB	P/EB EC	N PB	P/EC EB	N EA	P/EB EC
deviated	Li-loss layer removed		N N	N/ED N/ED	P/EA P/EA	N/EC N/EC	P/EA P/EA	P/EC P/EC	P P	P/EB P/EB
	Suspending horizontally		N N	N/EC N/EC	P P	P/EB P/EB	N N	N/EC P/EC	N/EA N	P/ED P/ED
	Exposure time	96h	E/PA E/PA	EB/C EC	PC PC	EB/C EC	PC PC	EC EC	EA/B EA/B	EB/C EC
		168h	E/PB E/PB	EC EC	PC PC	EC EC	PC PC	EC EC	EB EB	EC EC
	Solution temp. (35°C)		EA	EB	PA	PB	PA	EB	PA	EC

The corrosion attack of Al-Li series alloys exposed for the standard period of 48 hours was relatively slight. For example, little attack was revealed in 2090-T3, 8090-T3 etc. Even at 168 hour exposure, type of corrosion of Al-Li T3 alloys remained in pitting and no exfoliation corrosion took place at all.

From the above, it is found that this modified EXCO test solution is too mild for Al-Li series alloys to reveal exfoliation susceptibility.

3.3 ASSET Test

The summary of test results of ASSET test is listed in Table 3. Corrosion type of Al-Li series alloys was so almost all pitting that exfoliation corrosion occurred only in a few cases. The corrosion attack in 2024-T351 and 7075-T651, however, was exfoliation type.

Thus, ASSET test is not suitable to assess the susceptibility to exfoliation corrosion of Al-Li series alloys.

TABLE 3 SUMMARY OF RESULTS FOR ASSET TEST

Test condition			2090		2091		8090		2024	7075
			T3	T8	T3	T8	T3	T8	T351	T651
Standard (as specified)			PB EC	PC PC	PB EB	PC PB	PC EB	PC PB	EC ED	EB ED
deviated	Li-loss layer removed		PB PB	PC PC	PB PB	PC PC	PC PC	PC PC	EC EC	EC EB
	Suspending horizontally		PA PB	PC PB	PB PB	PC PC	PB PC	PC PC	EC EC	EC ED
	Exposure time	12h	EB EB	PC PC	EB EB	PC PC	EB EB	PC PC	ED ED	EC EC
		48h	EB EC	PC PC	EB EC	PC PB	EB EC	PC PB	ED ED	ED ED
	Solution temp. (35°C)		EB	PB	EA	PC	PA	PA	EC	ED

3.4 MASTMAASIS Test

Fig.2 shows the appearance and cross-section microstructure of the 2091-T8 tested under the standard condition in accordance with ASTM G 85. Judging from the appearance, the corrosion attack in 2090-T3, 2091-T8, 8090-T3, 8090-T8 and 7075-T651 was exfoliation type and the others are pitting. According to observation of cross-section microstructure, however, the type of corrosion was a mixed mode of pitting and exfoliation-corrosion.

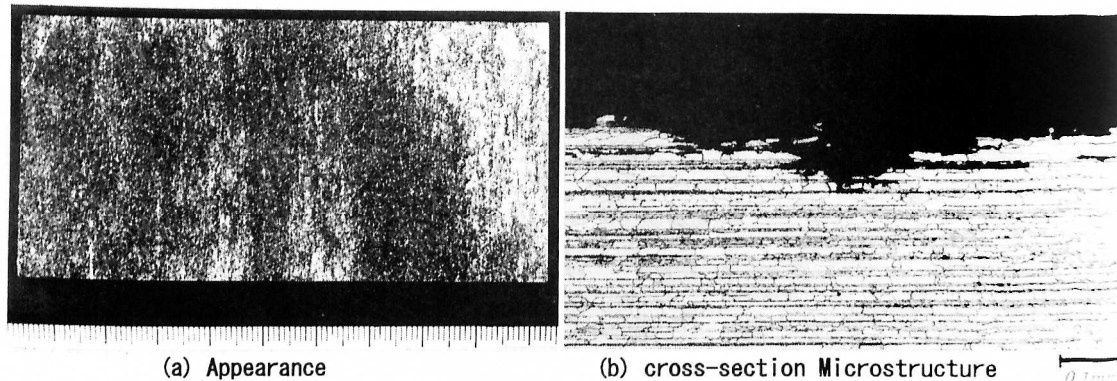


FIG. 2 APPEARANCE AND CROSS-SECTION MICROSTRUCTURE OF 2091-T8 TESTED UNDER THE STANDARD CONDITION. (MASTMAASIS TEST)

The summary of test results based on observation of cross-section microstructure evaluation is listed in Table 4. Most of the type of corrosion was pitting excepting exfoliation corrosion in 7075-T651.

As a result, the tendency in corrosion obtained by MASTMAASIS test is almost pitting corrosion for Al-Li series alloys. MASTMAASIS test is not available for evaluating the susceptibility to exfoliation corrosion in Al-Li series alloys as with ASSET test.

TABLE 4 SUMMARY OF RESULTS FOR MASTMAASIS TEST

Test condition		2090		2091		8090		2024	7075
		T3	T8	T3	T8	T3	T8	T351	T651
Standard (as specified)		E	P	P	P	P	P	E/P	E
deviated	Li-loss layer removed	P	P	P	P	P	E	E	E
	Exposure time (1 week)	P	P	E	P	P	P	E/P	E

4. CONCLUSION

- (1) EXCO test according to ASTM G34 is suitable to assess the relative exfoliation corrosion susceptibility in Al-Li series alloys. Exposure for 48 hours is sufficient to distinguish the corrosion type and rating.
- (2) Results of ASSET test per ASTM G66 and MASTMAASIS test per ASTM G85 show the pitting corrosion for Al-Li series alloys. Thus these tests are not suitable to assess the exfoliation corrosion in Al-Li series alloys.
- (3) The Li-loss layer in the surface region shows some effects on the exfoliation corrosion susceptibility of Al-Li series alloys. Higher test temperature of test solution relatively accelerates the corrosion attack in Al-Li series alloys.