CoA Memo No. 166

The College of Aeronautics

Department of Materials



# Assessment of Blasted Surfaces

Progress Report to Sponsors in the Surfacing Division,
The Welding Institute

by

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# THE INFLUENCE OF GRIT BLASTING ON THE BOND STRENGTH OF FLAME SPRAYED ALUMINIUM COATINGS

Report to Sponsors

## INTRODUCTION

Since the last report to sponsors circulated in mid-1967, considerable concern has been felt over the wide scatter of results. The scatter in results between specimens produced in a single batch was generally comparatively small, in the order of 15%, but occasional results occurred which showed much greater differences from the average. Identical experiments carried out in different batches exhibited rather more scatter than within single batches. Similar scatter was noted in the results of other workers[1,2]. Additionally, many blasting variables which had previously been considered important by metal sprayers appeared to have very little effect on the bond strength of flame sprayed aluminium coatings on mild steel. Consideration of scatter in results suggested four possible reasons for this scatter.

(i) inherent scatter due to the heterogeneous nature of the process.
(ii) variation in the preparation of flame sprayed test pieces.
(iii) residual stresses in the sprayed coating.
(iv) adhesive penetration of the coating.

The spraying process involves a number of operations most of which are performed manually so that some scatter in results is inevitable. Careful control and some automation of the grit blasting and metal spraying processes was adopted at an early stage in the work and effected some improvement in general consistency but gave no reduction in the percentage scatter recorded above. That residual tensile stresses are generated in the coating and base metal surface due to shrinkage of the sprayed metal has been known for some time and it has been accepted that these stresses can result in distortion of the component or failure of the sprayed metal/base plate bond. Considerable difficulties exist in measuring these stresses, however, and very little information is available on the magnitude of these stresses and none on their variation. The problem had been discussed by the Research Panel and it was decided that the variation of residual stresses was likely to be small at low coating thicknesses, for example between 0.002 in. and 0.005 in. The question of adhesive penetration of the spray coating was also considered by the Research Panel at an early stage in the work, since it was recognised that adhesive penetration through the coating to the bond interface could give erroneous results. However, no adhesive penetration was found in the metallographic examination of spray coatings made in 1966 and it was thereupon considered that its effects were insignificant. The possibility of adhesive penetration was raised again in mid-1967 shortly after the circulation of the last Progress Report and the problem was re-investigated. These results are reported below.

In an attempt to reduce the scatter in bond strength testing and to

indicate the possible causes of scatter a number of steps were taken. Control and standardisation of blasting, spraying and specimen preparation was tightened still further and three different adhesives (the one used before and adhesives recommended by an adhesive manufacturer and a metal sprayer) were used for different series of bond strength tests. Finally, tests were initiated to determine the variation of bond strength with coating thickness, a variable which had not previously been investigated since a coating thickness of 0.003 in-0.005 in. had been selected as representative of current industrial practice. The results of these investigations are reported below.

Materials and Equipment

Identical materials have been used to those reported previously. Some additional blasting work has been carried out using S204 round shot and G07, G12 and G17 chilled iron angular grit.

In work reported previously the adhesive used for bond strength testing was exclusively Araldite* AV100. This adhesive has been used for much of the present work but some tests have also been made using Araldite AV133, which was recommended by the manufacturers as being more viscous than AV100, and EC2186, an adhesive produced by the 3Ms Company.

Experimental Procedure

The conditions for grit blasting and metal spraying were maintained as before, except where a specific variable was under investigation. The standard blasting conditions for the 3 in. diameter specimens were:

| | |
|---|---|
| Grit: | G24 chilled iron, angular |
| Air pressure: | 65 psi |
| Blasting angle: | 90° |
| Nozzle-Plate distance: | 4 in. |
| Blasting speed: | 18 i.p.m. |

The specimen was completely blasted in 3 passes. Standard recommended spraying conditions were maintained for all the work, and the 3 in. diameter steel surface was covered in 3 passes. Each pass deposited a coating of approximately 0.002 in. thickness so that several sets of passes were necessary to build up coating thicknesses greater than 0.002 in.

In view of the scatter obtained in bond strength tests care was taken to maintain the standard conditions and spraying was carried out within 1 hour of

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* Trade Name for adhesives manufactured by CIBA Ltd.

blasting. Storage and transfer of blasted samples was carried out in desiccators and sprayed samples were also stored in desiccators. In view of the effect of coating thickness on strength, the standard thicknesses used was increased to 0.010 in.

The bond strengths of coatings were measured using the standard pull-off test reported earlier [3] (Fig. 1). A number of modifications were tried at various times to increase the consistency of results but no improvement was obtained.

An alternative test [4] (Fig. 2) was also used in one series of experiments. The coating was sprayed on to the prepared flat face of a 1 in. diameter bar, drilled and tapped at the other end to allow insertion in a tensile testing machine. The coating was surface ground and a similar, uncoated bar bonded to it with an adhesive, care being taken to ensure alignment between the two bars. After curing of the adhesive the specimens were tested in a tensile testing machine. As with all tests for bond strength the load during testing was maintained perpendicular to the coating by means of universal joints. No improvement in consistency was found with this test compared to the standard test.

## Experimental Work and Results

### 1. Adhesive Penetration

As mentioned above, the penetration of adhesive had been considered at various times in the work but it proved difficult to find evidence of any significant penetration. Steps taken to aid detection of adhesives included the addition of fluorescent dyes to the adhesives and the taking of a variety of sections of spray coatings and substrates. Examination of these sections showed that obvious penetration was extremely small with Araldite AV100, the deepest penetration observed being somewhat less that 0.003 in. and the area of the coating penetrated to this depth being very small (of the order of 1%)

Initially, it was accepted that these results proved that adhesive penetration was not a significant factor in influencing bond strength, but subsequent tests showing marked variation of bond strength with thickness led to a further examination using two other adhesives. The use of Araldite AV133 was advocated at one time since it was claimed to be more viscous than AV100. However, examination of spray coatings on which AV133 had been placed indicated rather more penetration than had been formed with AV100 and the use of this adhesive was discontinued. A third adhesive, EC2186 produced by 3 Ms was also used and, since it gave very high bond strengths, penetration of the coating was suspected. To aid detection, a quantity of fluorescent dye was again added to the adhesive and samples prepared for examination with different coating thicknesses up to 0.040 in. Although the total area of penetration was slight, penetration persisted to a depth of 0.009 in. in two samples.

The determination of the variation of bond strength with thickness

(reported below) led to a further examination of the penetration of aluminium spray coatings by AV100. Slugs were bonded with dyed AV100 on to 0.010 in. coatings on two surfaces blasted with new grit at an angle of 10°. The coatings were then pulled from the base plate and the undersides examined in ultra-violet light. In both cases penetration of the coating had occurred over less than 1% of the total area. In one case penetration had occurred at two points only but in the other sample a small crazed network was visible. This was the greatest depth of penetration observed and further tests are being carried out on these and other samples to gain further information.

2. Coating Thickness

All results indicate that the bond strength falls with increase in coating thickness to a minimum value of between 1000 psi and 2000 psi. The scatter in results was not influenced by testing techniques but the choice of adhesive did influence the strength measured for the thinner coatings.

All results using AV100 adhesive (a total of four series comprising 140 tests) are shown in Fig. 3. A considerable fall in bond strength occurred from 0.002 in. to 0.004 in. but from 0.006 in. to 0.040 in. the strength remained steady. The great majority of results lie between 1200 psi and 1900 psi with an average of approximately 1500 psi. Identical results were found when AV133 adhesive was used, although far fewer tests were made.

Bond strength results when EC2186 adhesive was used are shown in Fig. 4. The strength measured fell steadily as the coating thickness increased from 0.002 in. to 0.015 in. but remained steady from 0.020 in. to 0.040 in. Fewer results were obtained with this adhesive but the scatter band of results above 0.015 in. lay between 1400 psi and 2000 psi with a mean value of about 1650 psi.

The results obtained when the alternative test was used were very similar (Fig. 5).

As a result of these tests it was decided to increase the coating thickness for standard tests to 0.010 in. and this has been used for all the work reported below, except where stated.

3. Blasting Angle

The effect of blasting angle on bond strength is shown in Fig. 6 for new G24 grit, used G24 grit and well-worn grit. The results invalidate those found previously for thinner coatings and show a marked dependence on blasting angle. Further results for different grit sizes and S240 round shot are given in Fig. 7.

4. Nozzle-Plate Distance

Separations between 3 in. and 24 in. have been examined and all bond strength results lay well within the scatter band normally obtained (Fig. 8) The results obtained with finer grits lay within the same scatter band as G24 grit but results with round shot are considerably lower (Fig. 9)

5. Blasting Pressure

Results have been obtained between 85 psi and 30 psi, the minimum pressure at which the blasting cabinet would function. There was a slight indication that bond strength fell with pressure (Fig. 10) but more results would be needed to confirm this trend. Again, finer grit gave no different results to G24 grit but results with round shot were much lower and a slight dependence of bond strength of blasting pressure was indicated (Fig. 11)

6. Grit Condition

Three conditions of grit have been generally used throughout this work:

(i) brand new chilled iron angular grit, used for no more than 30 minutes. i.e. 8 cycles through the blasting cabinet.

(ii) G24 grit used for a minimum period of 50 minutes and showing some signs of rounding of the angular edges.

(iii) Well-worn dusty grit (probably originally G39) supplied as unfit for further use.

External assessment of samples of these grits considered[5] that the used grit was evenly sized and had only minor rounding on some edges. There was no obvious reduction in size and it appeared to be in excellent condition. The well-worn grit was reported to be less uniform in size than the new or used grit with considerable rounding of edges, particularly of the smaller particles. An estimated quarter (by volume) of the sample was of an average particle-mass equivalent to only about one-third of the mass of a new grit particle. However, the amount of dust in the "as supplied" sample had been completely removed by the filters in the blasting cabinet and this grit was considered useable for normal zinc or aluminium coatings. From the potential bond strength of sprayed aluminium on the grit blasted surface, the well-worn grit was assessed as giving 80% of the bond strength of brand new grit.

The effect of these three grits on the bond strength is shown in Figs. 6 and 10. No real difference can be distinguished between the brand new and used grit but the well-worn grit (with dust content removed) led to lower bond strengths.

Some further work using finer grits and round shot (Figs. 7, 9, 11) is at present underway. The finer grits behave identically to G24 grit at least in the new condition but round shot gives markedly lower bond strengths.

7. Delay Between Blasting and Spraying

The effect of delaying spraying after blasting for periods of 4 hours to 28 days is shown in Fig. 12 for specimens stored in air and a desiccator. All the results fall within the scatter band experienced in standard tests where spraying was carried out within 1 hour of blasting, so that no real effect could be distinguished. Further samples were stored on the laboratory roof after blasting and were submitted to heavy rain in the first few hours. Specimens sprayed after periods of 4, 24 and 48 hours gave low bond strengths between 325 and 625 psi and after this period a coating would not adhere to the baseplate.

8. Delay Between Spraying and Testing

These tests (Fig 13) supported previous results and all results fell within the expected scatter band, indicating no effect.

9. Effect of Metallurgical Structure

In order to check this effect samples of $1\frac{1}{2}$ in. diameter carbon steel rod were produced in the as rolled (Hv 220), oil quenched from 850°C (hv 316) oil quenched and tempered at 550°C (Hv 270) and furnace cooled from 850°C (Hv 187) conditions, giving a range of metallurgical microstructures. However, the bond strength of coatings on these different microstructures showed no significant differences.

10. Effect of Base Plate Thickness

Earlier work had shown a marked difference in bond strength between sprayed coatings on $\frac{1}{2}$in. thick specimens cut from 3" diameter bar and $\frac{1}{4}$ in. thick hot rolled mild steel plate. Examination of the two base plate materials showed little metallurgical difference although the bar was obviously higher in carbon content and had more pearlite in its microstructure but, as noted above, further tests showed no obvious metallurgical effects. The suggestion was then made that the difference might be due to either residual stress generated in the specimen during blasting or spraying, or to bending of thinner specimens during testing. In order to investigate these suggestions, spray coatings were produced on bar specimens of thicknesses 2 in., 1 in., $\frac{3}{4}$ in., $\frac{1}{2}$ in. and $\frac{1}{4}$ in. cut from the 3" diameter bar. Other coatings were sprayed on to 2" thick bars which were then reduced to thicknesses of 1 in., $\frac{3}{4}$ in., $\frac{1}{2}$ in. and $\frac{1}{4}$ in. Bond strength results

for these tests are given in Fig. 14. Although there appears to be a very slight fall in bond strength for the thinnest base plates the difference is not significant and lies within the scatter band.

11. Effect of Masking

In order to fully coat 3 in. diameter specimens, three passes with the spray gun were necessary. It was suggested that the edges of each pass of spray coating might have inferior bond strength due to a longer travel path and oblique angle of impact on the base. In order to check this suggestion base plates were masked with tape to ensure that only the central deposits from the spray gun were utilised. No difference in bond strength was observed.

12. Surface Preparation Methods other than Blasting

Several Tests have been carried out to look at the influence of base surface condition upon the bond strength. These tests were made with coatings of 0.003 in. to 0.005 in. thickness so that they must be interpreted cautiously but several interesting facts were observed. From the results (Table 1) it may be noted that machined and scratch brushed surfaces were inadequate for any bond to be formed but that bonding could occur if the machined surface was deeply etched with an acid or oxidised by heat treatment.

13. Heat Treatment

The effect of heat treatment at various temperatures in air before and after spraying is shown in Table 11. Again tests were carried out on samples with thin spray coatings so that the results can only be viewed qualitatively. Heat treatment of the base surface before spraying at temperatures of 300°, 400° and 450°C gave a marked improvement in bond strength but the effect of treatment at 200°C and 600°C was less marked. Heat treatment at 450°C after spraying, gave a further improvement in all cases.

Work at B.I.S.R.A.[5] on roll bonded aluminium on steel had indicated that improvements in bond strength by similar heat treatment was due to the formation of a spinel at the interface. This appeared to be the reason for the improvement in bond strength of the aluminium spray coatings but as a check a series of samples were prepared in which the bond strength was improved by heat treatment in air before and after spraying and then followed by a heat treatment at 450°C for ½ hour in a hydrogen atmosphere to reduce any oxide bond. This treatment did, in fact, reduce the bond strength to the levels obtained without oxidising heat treatment indicating that improved bond strengths were due to an oxide formation at the interface.

14. Residual Stress

A number of attempts have been made to investigate the level and variation of residual stresses in spray coatings but without success. Two facts

which may be significant are the uniform bond strengths obtained for coating thicknesses of 0.006 in. to 0.040 in. and for the 0.020 in. thick coatings reduced in size between 0.015 in. and 0.006 in. Additionally, spray coatings of up to 0.25 in. thick have been produced without spalling of the coating.

15. Position of Failure

In the majority of cases failure on bond testing has been entirely at the coating - base interface (termed "adhesion" failure) but in some cases some failure within the coating occurred ("cohesion" failure). Results will be presented in the final report but in general cohesion failure is never met below a bond strength of 1000 psi and rarely at 1500 psi. At higher bond strengths with coating thicknesses above 0.006 in. it becomes more prevalent but rarely exceeds 15% of the test area.

## DISCUSSION OF RESULTS

It is intended to leave main consideration of the results until the final report but a few brief comments can be made.

A small amount of adhesive penetration does occur but it appears that this has a marked effect on bond strength measurements on thin coatings, possibly by forming some sort of "composite". The influence of residual stresses in the coating cannot be ruled out although the static level of bond strength between 0.006 in. and 0.040 in. indicates that it is of less importance than adhesive penetration. No information is available on the significance of residual stresses in the base material.

On the results of the present work, grit size appears to have little effect but grit condition is very important. The transition from brand new angular grit to worn grit results in a fall in bond from about 1500 psi to about 1100 psi whilst the use of round shot gives a strength of below 300 psi. This could well be the lower limit of bond strength that can be obtained by blasting, provided that clean grit is used. Earlier work indicates that dusty grit could give inferior results.

The significance of the other blasting variables, with the exception of angle, appears to be minimal and all apparent trends lay within the scatter band. It should be emphasized that results have been assessed on bond strength only. Thus, higher blasting pressures are often used not to improve bonding but to increase the rate of blasting: this aspect has not been investigated.

Blasting angle does appear important, low results being obtained when angles of 30° or less are used. The effect might well be more marked as grit condition deteriorates.

Grit blasting is obviously the best method of preparing surfaces for spraying, but deep etching gives a more or less suitable surface and might prove preferable where blasting would cause excessive distortion. Combined with he at treatment at 450°C good bond strengths should be possible.

The effect of heat treatment in improving bond strength has been shown and might have commercial significance. The mechanism is probably similar to that occurring on roll bonding aluminium on steel but whether this involves forming a spinel is debatable. A simple oxide to oxide bond would appear equally likely .

## REFERENCES


1. Ballard, W. E., Metal Spraying and the flame deposition of ceramics and plastics, Griffin, London, (1963).

2. Smith, R. S. and Stephenson, N., British Welding Journal, 8, (4), 161, (1961)

3. Apps, R. L., The effect of grit blasting on the bond strength of sprayed aluminium coatings on mild steel, Second Divisional Conference of the Metal Spraying and Plastic Coating Division, Institute of Welding, London (1967)

4. Evaluation methods and equipment for flame sprayed coatings, METCO INC., New York, (1963).

5. Gerhold, E. A., Private communication

6. Bullough, W., Bonding of aluminium foil and powder to steel strip, JISI, Jan. 1967, p.6.

TABLE I

THE EFFECT OF SURFACE PREPARATION METHODS OTHER THAN GRIT BLASTING.

| | Bar Material | | Plate Material | |
|---|---|---|---|---|
| Surface Preparation | Bond Strength psi | Comments | Bond Strength psi | Comments |
| As received with scale | - | No test | 0 | No spray adhesion |
| As machined | 0 | No spray adhesion | 0 | No spray adhesion |
| As machined and heated to 450°C for 30 mins. | 1165 | Good adhesion to base | 352 | Adhesion to base occurred |
| As machined and heated to 700°C for 30 mins. | 535 | Slight peeling of coating at edge of specimen. | 567 | Spray coating adhered to base. |
| Scratched, brushed | 0 | No spray adhesion. | 0 | No spray adhesion |
| As machined and etched in 10% nital for 2 mins. | 0 | Good micro etch of surface. No spray adhesion. | 0 | No spray adhesion even after 2 hrs. etching. |
| As machined and etched in 10% $H_2SO_4$ for 15 hrs. | 2045 | Surface deeply attacked by $H_2SO_4$. Good spray adhesion to base. | - | No test. |

TABLE II

THE EFFECT OF HEAT TREATMENT ON BOND STRENGTH

| Heat Treatment | Bond Strength psi | Location of Failure | | |
|---|---|---|---|---|
| | | Adhesive Failure, % | Adhesion Failure, % | Cohesion Failure, % |
| Bar grit blasted, heated for 20 mins. at 200°C and air cooled before spraying | 1780 | 0 | 85 | 15 |
| | 2355 | 2 | 78 | 20 |
| | 2325 | 0 | 85 | 15 |
| Heat treatment after blasting and before spraying at 200°C (as above) followed by heating at 450°C for 20 mins. after spraying. | 3900 | 10 | 40 | 50 |
| Bar grit blasted, heated for 20 mins. at 300°C and air cooled before spraying | 3860 | 10 | 40 | 50 |
| | 4220 | 10 | 15 | 75 |
| | 4180 | 10 | 0 | 90 |
| Heat treatment after blasting and before spraying at 300°C (as before) followed by heating at 450°C for 20 mins. after spraying. | 3810 | 50 | 0 | 50 |
| Bar grit blasted, heated for 20 mins at 400°C and air cooled before spraying | 3770 | 25 | 5 | 70 |
| | 3000 | 5 | 35 | 60 |
| | 3450 | 0 | 10 | 90 |
| Heat treatment after blasting and before spraying at 400°C (as above) followed by heating at 450°C for 20 mins. after spraying | 5275 | 15 | 10 | 75 |
| Bar grit blasted, heated for 20 mins. at 450°C and air cooled before spraying | 4360 | 40 | 50 | 10 |
| | 4825 | 25 | 55 | 20 |
| | 4690 | 20 | 20 | 60 |
| Heat treatment after blasting and before spraying at 450°C (as above) followed by heating at 450°C for 20 mins. after spraying | 5220 | 80 | 10 | 10 |

TABLE II (cont.)

| Heat Treatment | Bond Strength psi | Location of Failure Adhesive Failure, % | Adhesion Failure, % | Cohesion Failure |
|---|---|---|---|---|
| Bar grit blasted, heated for 20 mins. at 600°C and air cooled before spraying | 2760 | 15 | 80 | 5 |
| | 3950 | 10 | 85 | 5 |
| | 3620 | 0 | 95 | 5 |
| Heat treatment after blasting and before spraying at 600°C (as above) followed by heating at 450°C for 20 mins. after spraying. | 4190 | 5 | 90 | 5 |

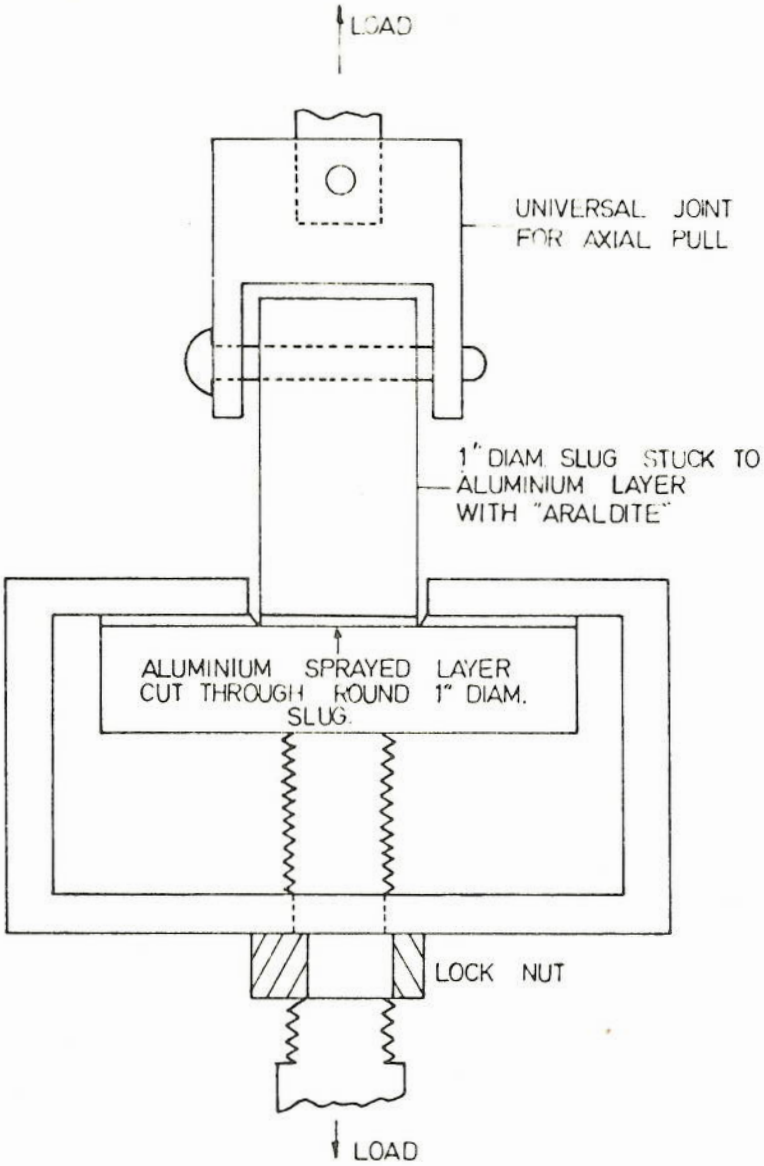


FIG. 1. STANDARD PULL-OFF TEST FOR BOND STRENGTH

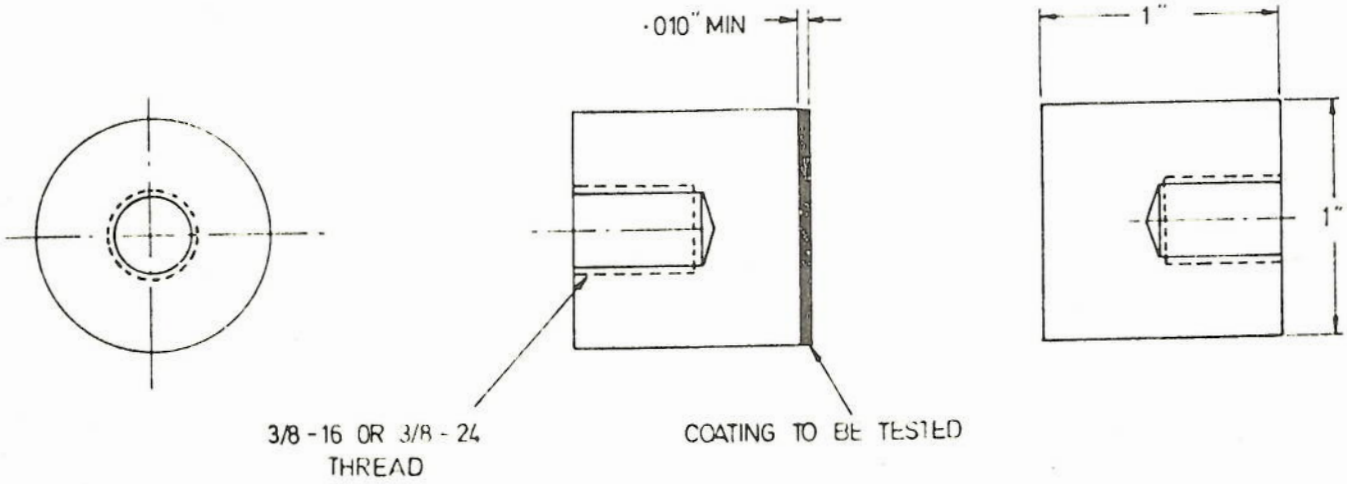


FIG. 2. ALTERNATIVE TEST FOR BOND STRENGTH.

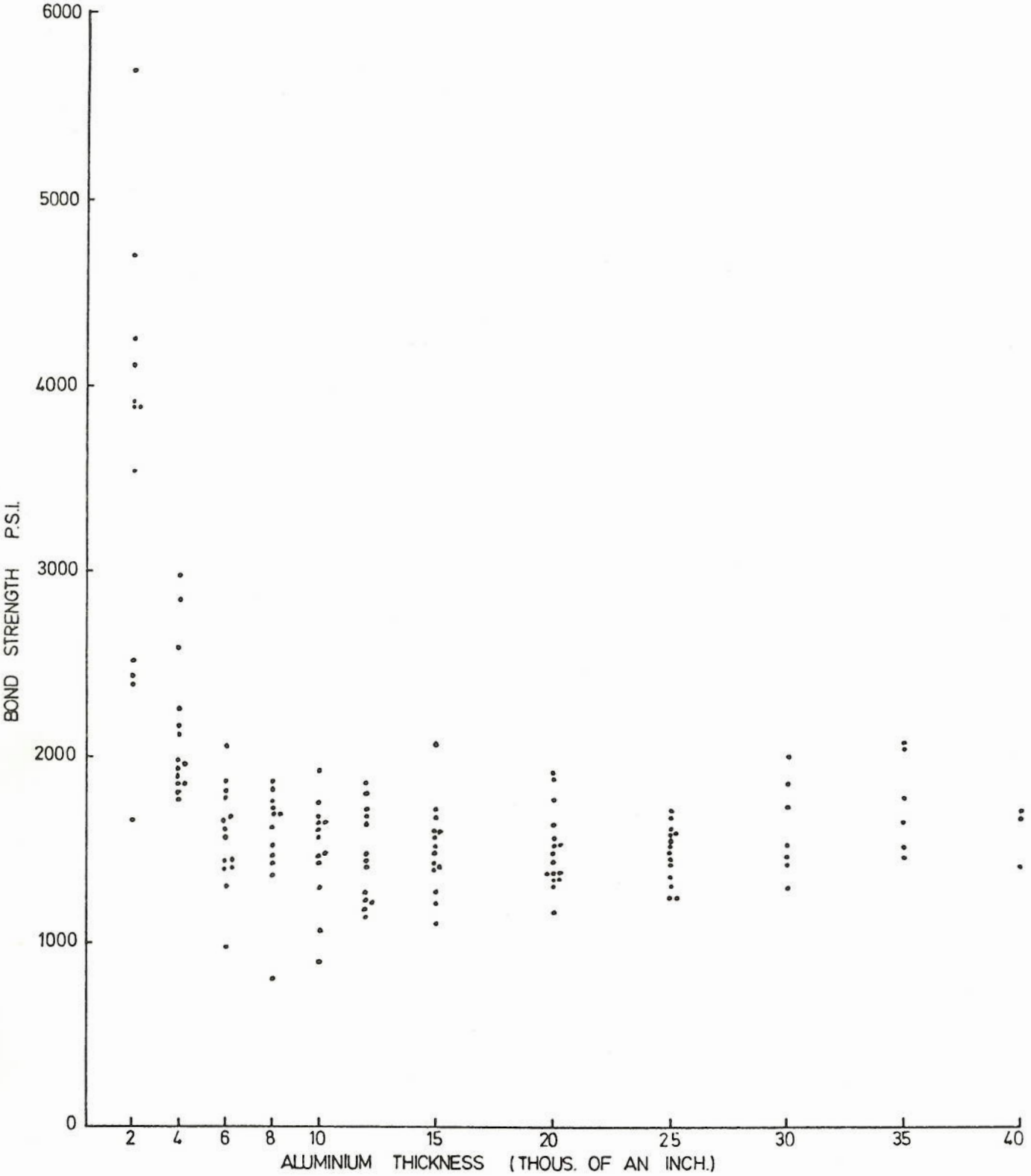


FIG. 3. VARIATION OF MEASURED BOND STRENGTH WITH COATING THICKNESS (AV100)

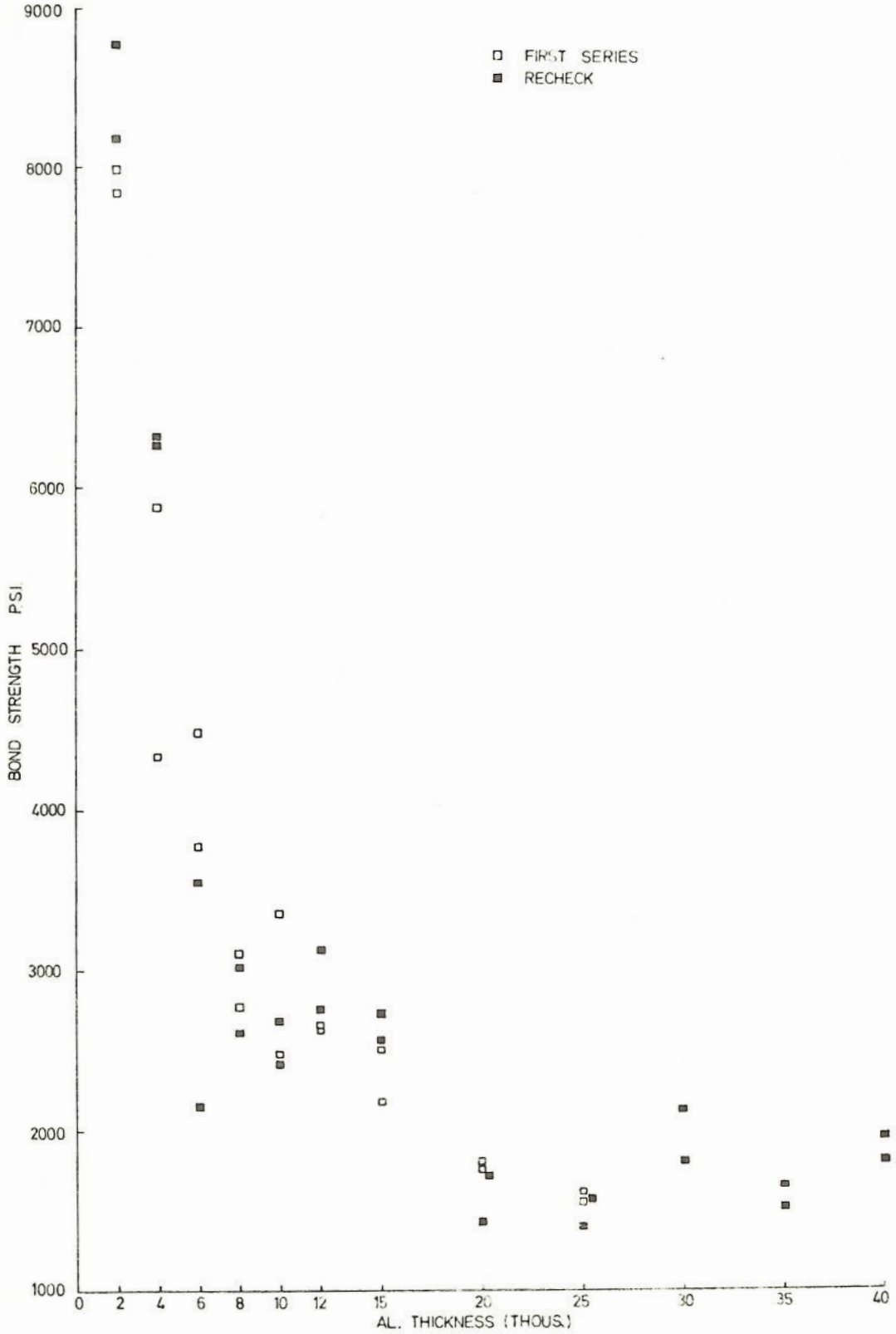


FIG. 4. VARIATION OF MEASURED BOND STRENGTH WITH COATING THICKNESS (EC 2186)

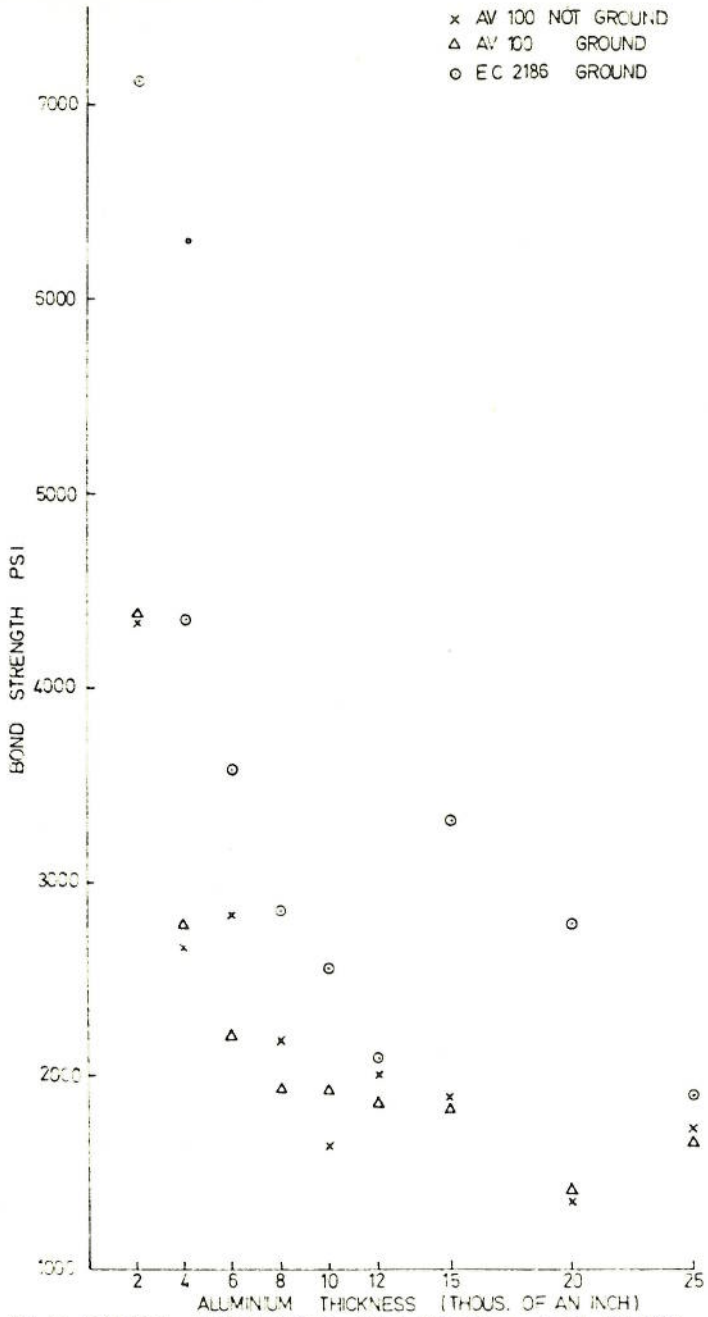


FIG 5. VARIATION OF MEASURED BOND STRENGTH WITH COATING THICKNESS (ALTERNATIVE TEST METHOD).

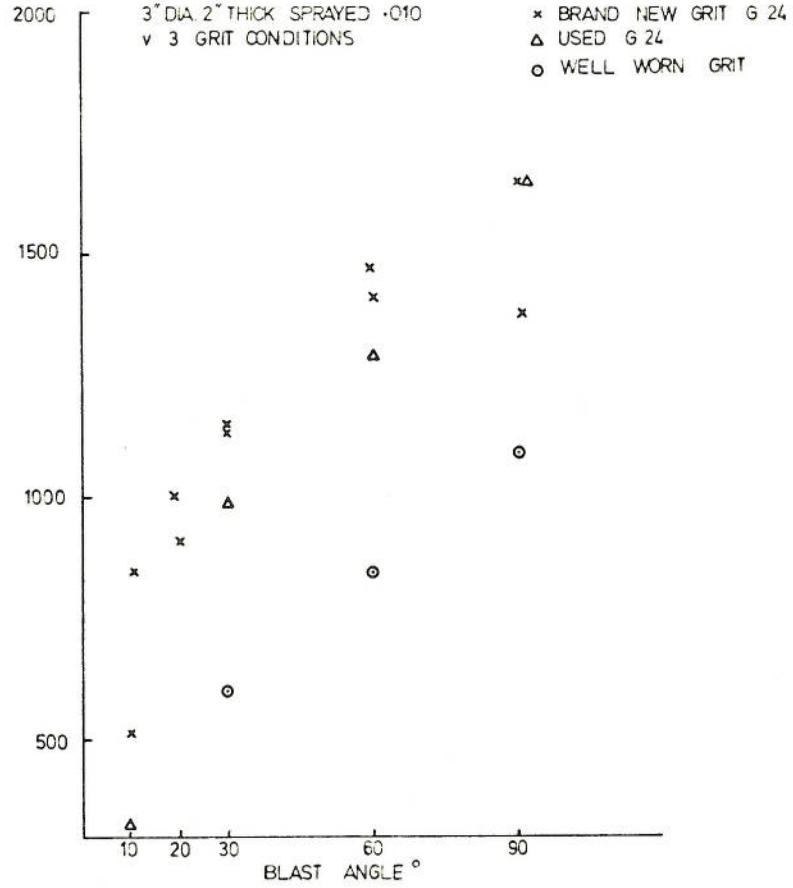


FIG 6. BOND STRENGTH VERSUS BLASTING ANGLE FOR NEW, USED AND WELL-WORN GRIT.

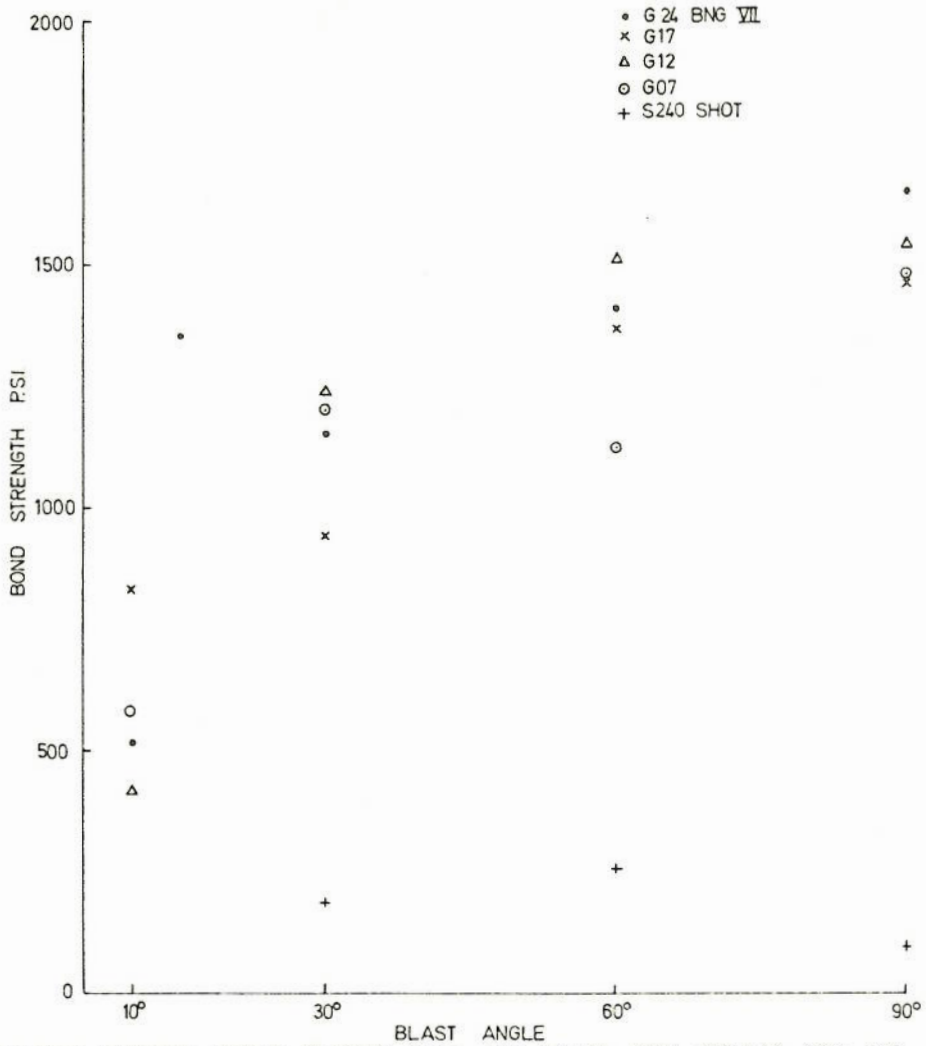


FIG 7 BOND STRENGTH VERSUS BLASTINC ANGLE FOR CHILLED IRON ANGULAR GRIT AND ROUND SHOT.

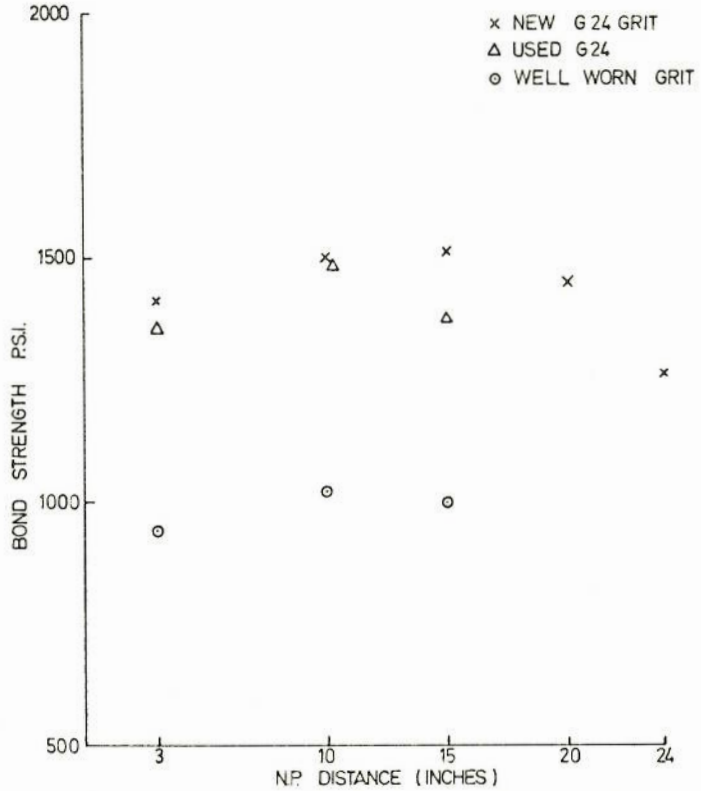


FIG. 8. VARIATION OF BOND STRENGTH WITH BLASTING NOZZLE-PLATE DISTANCE.

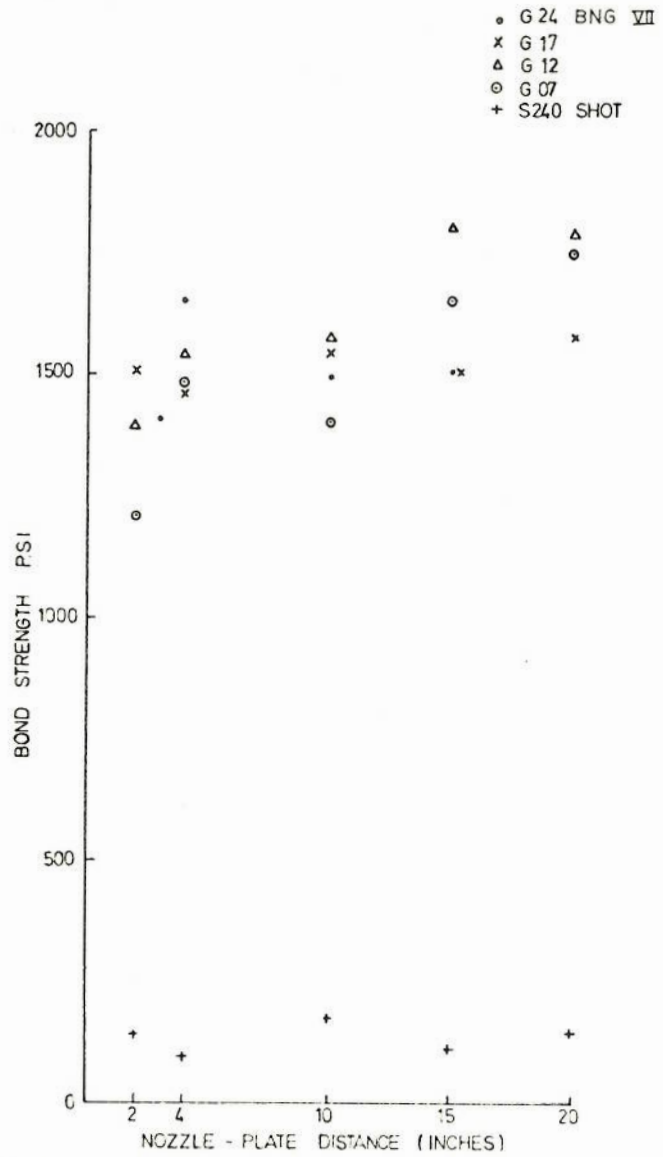


FIG. 9. VARIATION OF BOND STRENGTH WITH BLAST ANGLE FOR FINER GRITS AND ROUND SHOT.

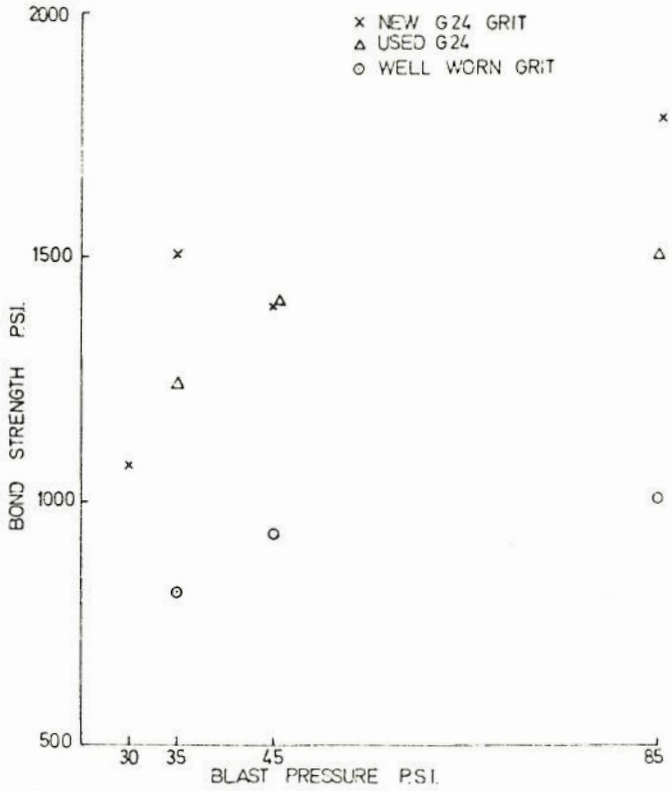


FIG. 10 BOND STRENGTH VERSUS BLASTING PRESSURE.

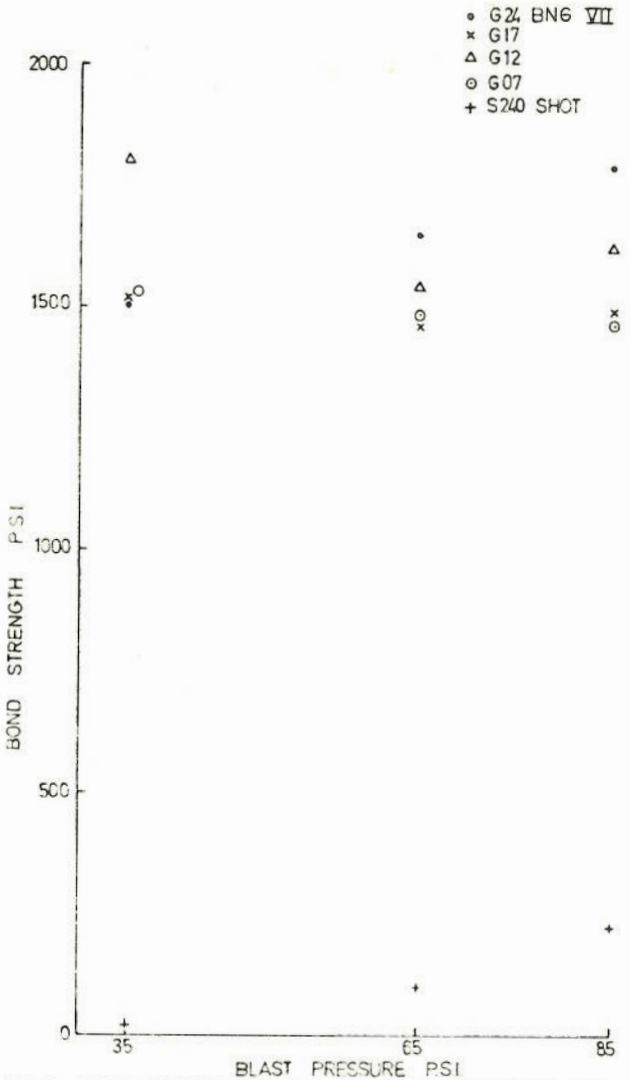


FIG. 11 BOND STRENGTH VERSUS BLASTING PRESSURE FOR VARIOUS GRIT SIZES AND ROUND SHOT.

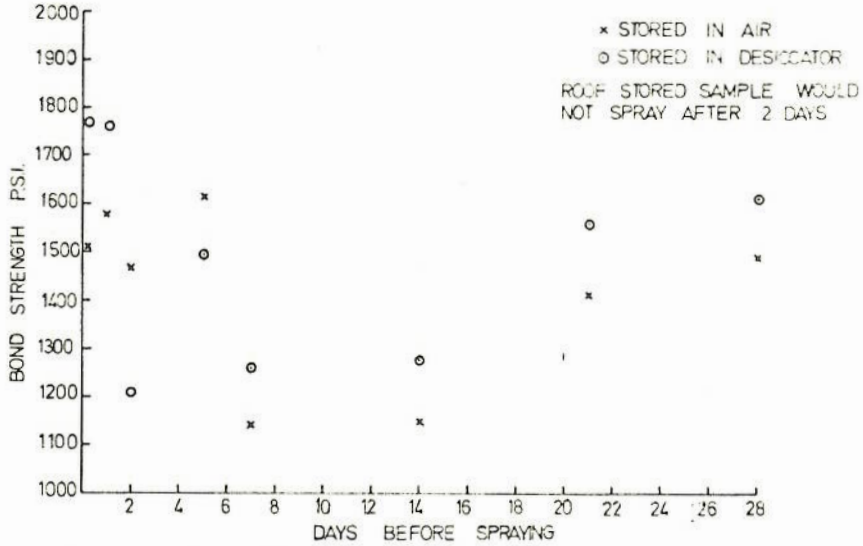


FIG. 12 EFFECT OF DELAY BETWEEN BLASTING AND SPRAYING UPON BOND STRENGTH.

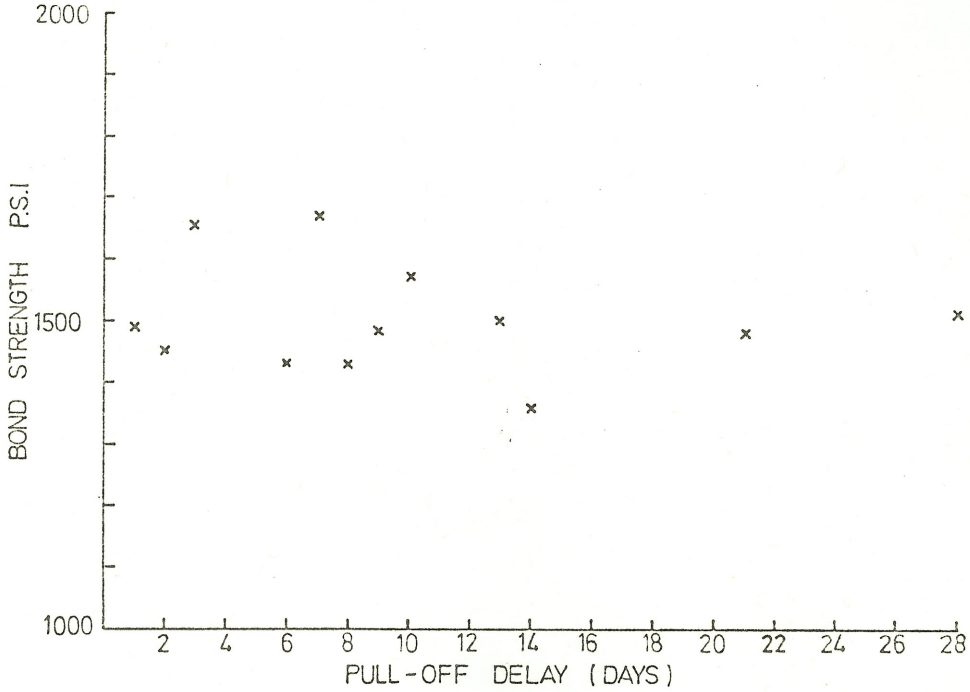


FIG. 13. EFFECT OF DELAY BETWEEN SPRAYING AND TESTING ON BOND STRENGTH.

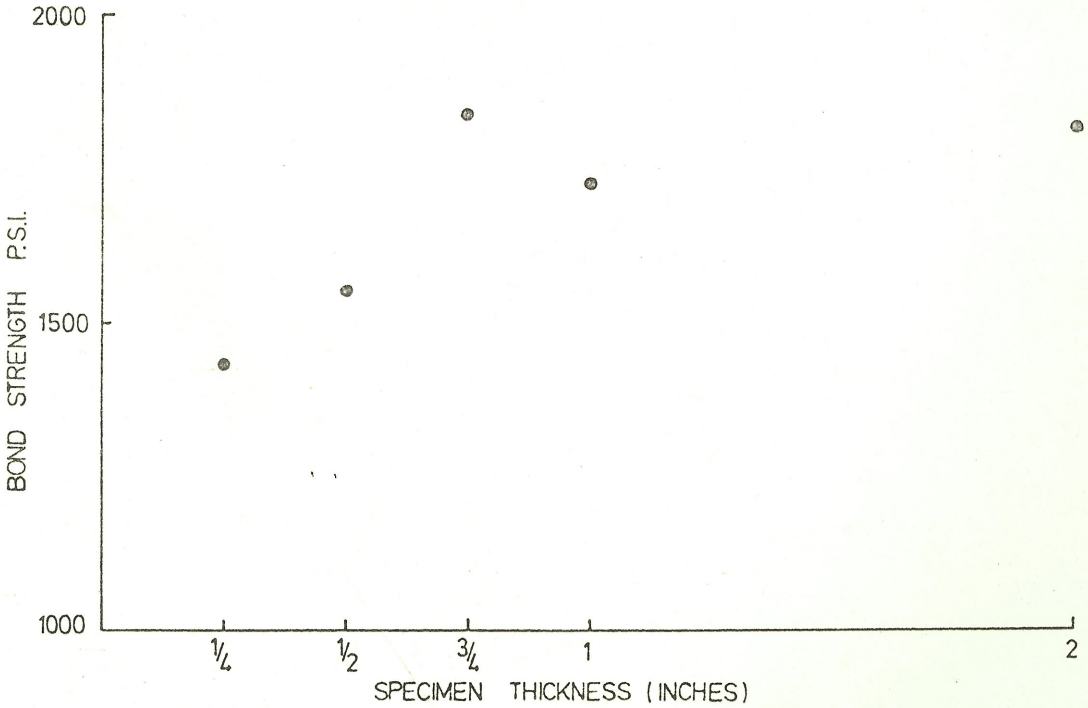


FIG. 14. EFFECT OF SPECIMEN THICKNESS ON BOND STRENGTH.