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(54) **RADIOGRAPHIC ASSEMBLAGE**

RADIOGRAPHISCHES ANORDNUNG

ASSEMBLAGE RADIOGRAPHIQUE

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- **RESEARCH DISCLOSURE, vol.218, no.41, June 1982, HAVANT GB pages 223 - 225 P.DE MAAYER ET AL. 'Ultraviolet-radiation emitting X-ray intensifying screens'**

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Description

[0001] The present invention concerns a radiographic assemblage comprising at least one X-ray intensifying screen and a radiographic film which is sensitive to actinic radiation. Such an assemblage enables very high quality radiographs to be obtained, even when a fast processing method is used.

[0002] In radiography, and particularly in medical radiography, radiographic assemblages are commonly used which comprise an intensifying screen and a photographic film consisting of a support covered on at least one of its two faces with a layer of silver halide emulsion.

[0003] The use of intensifying screens makes it possible to reduce the quantity of X-rays necessary to obtain a radiograph and consequently to reduce the quantity of X-rays absorbed by the patient.

[0004] These intensifying screens absorb the X-rays and re-emit electromagnetic radiation with a longer wavelength, the modulation of which is registered by the silver halide grains forming the radiographic emulsion.

[0005] The use of tabular-grain silver halide emulsions in radiographic products is known.

[0006] For example, US patent 4 639 411 describes a radiographic film which comprises an emulsion consisting of tabular silver halide grains and able to form a latent image when it is exposed to blue light. This emulsion preferably consists of tabular grains with a thickness of less than 0.5 µm and an aspect ratio greater than 5:1.

[0007] In order to optimise the absorption of blue light by the silver halide grains, sensitising dyes are adsorbed on the surface of the grains.

[0008] The radiographic film also comprises, interposed between the support and the layer of emulsion, a layer of silver iodide emulsion which absorbs blue light.

[0009] Such a radiographic film makes it possible to reduce the loss of sharpness due to the diffusion of the light through the support (cross-over).

[0010] Patent application WO 93/01521 describes a radiographic assemblage comprising an intensifying screen which emits in the ultraviolet wavelengths associated with a special radiographic film. These radiographic films consist of emulsions in which the proportion of chloride in the silver halide grains is at least equal to 50% molar and/or the silver halide grains are tabular grains with a thickness of less than 0.5 µm and preferably between 0.21 and 0.30 µm, and a mean aspect ratio greater than 2:1 and preferably between 4.0 and 5.5:1.

[0011] The examples in patent application WO 93/01521 describe a tabular-grain silver bromoiodide emulsion with a thickness of 0.2 µm and an aspect ratio of 5:1.

[0012] The intensifying screens associated with these radiographic films comprise a support covered with a layer of luminophores with an emission peak between 300 and 390 nm.

[0013] Such a radiographic assemblage enables good sensitivity and good image quality to be obtained even for a low exposure to X-rays.

[0014] In parallel to the development of more efficient radiographic assemblages, attempts are being made to process radiographic films by faster methods. The length of a traditional fast processing method is around 90 seconds; the length of the most efficient processing methods is around 45 seconds.

[0015] These fast processing methods are generally not very compatible with conventional radiographic films.

[0016] It is necessary to avoid water retention during the development, fixing and washing stages and to avoid physical defects which appear during drying. Consequently, the stage which limits a fast processing method is the film drying stage. This is why films intended to be processed in this way must be highly tanned.

[0017] The tanning level of the emulsions has a strong influence on the development of the silver, in particular with non-tabular silver halide grains. For example, when emulsions with non-tabular grains (referred to in the remainder of the application as "three-dimensional grains") are highly tanned, the covering power of these grains after development is very limited, which causes the sensitivity of the radiographic films to be greatly decreased.

[0018] It is therefore very useful to develop a radiographic assemblage which can be used in fast processing methods and which makes it possible to obtain quality radiographs with a low level of exposure to X-rays.

[0019] As is shown by the prior art cited above, the use of tabular-grain emulsions in radiographic assemblages is known. However, none of the known systems has the sensitometric advantages of the present invention.

[0020] The object of the invention is a Screen/Film radiographic assemblage which can be used with a fast processing method and which has an improved sensitometric performance as well as good image quality.

Figures 1 and 2 show the intrinsic sensitivity of grains with different morphologies as a function of the exposure wavelength.

Figure 3 shows the performance of the assemblage of the present invention in comparison with the known conventional assemblage.

[0021] The radiographic assemblage of the invention comprises (a) an X-ray reinforcing screen which emits mainly ultraviolet radiation and (b), associated with this screen, a photosensitive film comprising a tabular-grain silver halide emulsion. This radiographic system may be developed by fast processing methods.

[0022] Ultraviolet radiation (referred to hereinafter as "ultraviolet") is defined as the electromagnetic radiation with a wavelength between that of violet light and that of X-rays.

[0023] The radiographic assemblage of the invention comprises at least one X-ray intensifying screen and a radiographic film consisting of a support covered on at least one of its faces with a silver halide emulsion and is characterised in that at least 50% of the silver halide grains of the emulsion are tabular grains with an average thickness of less than 0.5 μm and preferably less than 0.2 μm, and an aspect ratio (R) between 5 and 25 and preferably between 10 and 20, and the X-ray intensifying screen comprises a support covered with a fluorescent layer comprising dispersed in a binder, a luminophore with a maximum emission as close as possible to the intrinsic sensitivity peak of the silver halide grains forming the radiographic film.

[0024] The aspect ratio (R) is the ratio of the equivalent circular diameter (ECD) to the average thickness of the tabular grains (e).

[0025] The emulsions used in the assemblage of the invention consist of grains of silver chloride, silver bromide, silver iodide or a mixture of these halides, in a binder.

[0026] The binder is a water-permeable hydrophilic colloid such as gelatin, gelatin derivatives, albumin, polyvinyl alcohol, polyvinyl polymers, etc.

[0027] These emulsions can be prepared in accordance with the precipitation techniques described in US patents 4.425.425 or 4.425.426.

[0028] According to one embodiment, monodisperse emulsions are used. The monodispersity of the emulsion is defined from the coefficient of variation (COV) which, expressed as a percentage, is equal to (σ/ECD).100.

[0029] The preferred monodisperse emulsions have a COV of less than 20% and preferably below 10%. These monodisperse tabular- grain emulsions can be prepared in accordance with the method described in US patent 5.210.013.

[0030] The emulsions used in the assemblage of the invention can be tanned in accordance with one of the methods described in US patent 4.425.426. The tanning agents which can be used are described in Research Disclosure, December 1989, No 308113, Section X.

[0031] In addition to the characteristics specifically described above, the emulsions may contain other compounds such as anti-fogging agents, stabilisers or antistatic agents. The radiographic film may comprise a top layer containing matting agents. This top layer or the sensitive layer may contain plasticisers or lubricants. These compounds were described in Research Disclosure, Vol 184, August 1979, No 18431.

[0032] These silver halide emulsions used in the assemblage of the invention are preferably chemically sensitised by means of sulphur and/or gold and/or selenium, in accordance with the conventional chemical sensitisation methods described in Research Disclosure, December 1989, No 308119, Section III.

[0033] The emulsions used in the assemblage of the invention may be spectrally sensitised. The conventional spectral sensitisation methods which can be used within the scope of the invention are described in Research Disclosure, December 1989, No 308119, Section IV.

[0034] As shown in Figure 1, the maximum intrinsic sensitivity of the tabular silver halide grains is, surprisingly, situated in the ultraviolet range; this sensitivity peak in the ultraviolet range is specific to tabular grains; it is not observed in non-tabular silver halide grains.

[0035] In addition, the intensity of the sensitivity peak varies with the aspect ratio of the tabular grains. The presence of such a sensitivity peak makes it possible to explain why tabular- grain emulsions have a sensitivity greater than or equal to that of three-dimensional grains, independently of the presence of spectral sensitising dyes (Fig 2).

[0036] The radiographic films used in the assemblage of the invention are therefore obtained in a smaller number of stages.

[0037] The films used in the assemblage of the invention can be developed using conventional development processes.

[0038] In addition, the radiographic films used in the assemblage of the invention are totally compatible with fast processing methods. Indeed, emulsions consisting of tabular grains with a high aspect ratio may be highly tanned without any impairment of their covering power being observed.

[0039] In one embodiment of the invention, the photographic film consists of a support able to transmit ultraviolet.

[0040] However, such a support may adsorb part of the ultraviolet passing through it. This adsorption of the support enables the cross-over to be reduced without having recourse to the additives commonly used for this purpose.

[0041] Within the scope of the invention, a support made of a polyester such as polyethylene terephthalate can be used.

[0042] The support is covered on at least one face, and preferably on both faces, with a silver halide emulsion with grains with a high aspect ratio according to the invention. The two faces of the supports may carry layers of identical or different emulsions.

[0043] The X-ray intensifying screen used in the assemblage of the invention comprises a support covered with a fluorescent layer comprising, dispersed in a binder, a luminophore having a maximum emission between 300 and 390 nm.

[0044] The X-ray intensifying screen used in the assemblage of the invention comprises one or more luminophores mixed in a binder. The luminophore or luminophores used have a maximum emission in the ultraviolet. This maximum emission must be as close as possible to the intrinsic sensitivity peak of the silver halide grains forming the radiographic film.

[0045] The luminophores which emit in the ultraviolet are, for example, thallium oxides such as $YTaO_4$, which may be activated by gadolinium, bismuth, lead or cerium atoms, or a mixture of these activators, the oxides of lanthane La_2O_2S or LaOBr, whether activated or not. The luminophores may consist of zirconium germanate and hafnium germanate crystals with a tetragonal scheelite structure and containing no titanium, as described in European patent application 552116.

[0046] These luminophores may be used alone or in a mixture.

[0047] The luminophores used in the assemblage of the invention have an emission peak between 300 and 390 nm and preferably between 310 and 360 nm.

[0048] The X-ray intensifying screens comprise a support covered with a fluorescent layer containing the luminophore or luminophores in the form of particles in a mixture in a binder. The size of the particles of the luminophores is generally between 0.5 and 10 µm and preferably between 1 and 20 µm.

[0049] The useful binders are chosen from organic polymers transparent to ultraviolet and to X-rays, such as vinyl alcohol and o-sulphobenzaldehyde acetal polymers, chlorosulphonated polyethylenes, bisphenol polycarbonates, copolymers of alkyl acrylate and methacrylate, or polyurethanes. Other binders which can be used within the scope of the invention are described in US patents 2.502.529, 2.887.379, 3.617.285, 3.300.310, 3.300.311 and 3.743.833 and in Research Disclosure, Vol 154, February 1977. The preferred binders are polyurethanes such as Estane®, Permuthane® and Cargill®.

[0050] The X-ray intensifying screen may contain, in addition to the fluorescent layer, a protective layer and a reflective layer.

[0051] The methods of manufacturing the screens and radiographic films are described in the Research Disclosure cited above, No 18431.

[0052] The radiographic assemblage of the invention comprises in general terms a pair of X-ray intensifying screens situated on each side of the radiographic film.

[0053] The combination of a film as described above and an X-ray intensifying screen which emits in the ultraviolet wavelengths enables a radiographic assemblage to be obtained which is more efficient than a conventional system equipped with a screen which does not emit in the ultraviolet wavelengths.

[0054] Such a radiographic assemblage makes it possible inter alia to expose the patient to smaller quantities of X-rays.

[0055] The present invention is illustrated by the following examples which show the sensitometric advantages of the invention in comparison with conventional radiographic assemblages.

EXAMPLES

[0056] The films used in the assemblage of the invention described in the following examples consist of an ESTAR® support covered, in this order, with a layer of silver bromide emulsion (21 mg/dm^2) and a top layer of gelatin (6.88 mg/dm^2). The film is tanned by means of bis (vinylsulphonylmethyl) ether, the content by weight of tanning agent being equal to 2.25% of the total dry gelatin contained in the film. These emulsions are monodisperse (COV of less than 10%) and chemically sensitised optimally by means of sulphur and gold, the quantity of sulphur being between 15,000 and 25,000 At/µm^2 and the quantity of gold between 7,500 and 12,500 At/µm^2.

[0057] The tabular silver bromide grains represent more than 90% of the total number of grains forming the emulsion.

EXAMPLE 1

[0058] This example is illustrated in Figure 1.

- Figure (1A) shows the spectral sensitivity curve obtained with X-OMAT-S® film, which consists of a three-dimensional grain emulsion.

- Figure (1B) shows the spectral sensitivity curve obtained from a tabular silver bromide grain emulsion (ECD = 2.40 µm, e = 0.125 µm, R = 19.2, COV = 6.6) which was sensitised with a blue-sensitive sensitising dye (maximum absorption 440 nm)

- Figure (1C) shows the spectral sensitivity curve obtained from a tabular silver bromide grain emulsion (ECD = 2.40 µm, e = 0.125 µm, R = 19.2, COV = 6.6) which was not chromatised.
- Figure (1D) shows the absorption curve of the support.

[0059] It can be seen that in the ultraviolet region the films consisting of tabular-grain emulsions have a sensitivity peak which does not appear with the X-OMAT-S® commercial film consisting of a three-dimensional grain emulsion. Consequently the sensitivity peak is not related to the chromatisation of the tabular emulsion.

<u>EXAMPLE 2</u>

[0060] This example is illustrated in Figure 2. The various figures are obtained with the emulsions described in the following table:

REFERENCE	E.C.D. µm	THICKNESS µm	COV %	ASPECT RATIO
2A	2.40	0.125	6.6	19.20
2B	2.63	0.085	7.6	30.94
2C	3.12	0.140	3.9	22.29
2D	1.45	0.60	7.9	2.42
2E	1.96	0.49	6.2	4.00

[0061] Figure 2 shows that the sensitivity peak varies with the aspect ratio of the tabular grains.

<u>EXAMPLE 3</u>

[0062] The tabular grain emulsions used in the assemblage of the invention described in the following table were used to produce radiographic films as described in the introductory part of the examples.

REFERENCE	ECD µm	THICKNESS µm	ASPECT RATIO	RELATIVE SPEED AT 330 nm	RELATIVE SPEED AT 430 nm
1	0.85	-	1.00	100	90.2
2	1.45	0.60	2.42	105.9	48.1
3	1.96	0.49	4.00	134.3	47.0
4	2.39	0.30	7.97	185.4	58.5
5	2.43	0.28	8.68	221.3	62.2
6	2.69	0.225	11.96	209.4	54.6
7	2.88	0.23	12.52	225.9	47.8
8	2.40	0.125	19.20	173.8	45.8
9	2.82	0.131	21.53	199.1	56.8
10	3.07	0.142	21.62	131.8	29.2
11	3.12	0.14	22.29	139.3	34.4
12	3.65	0.135	27.04	127.6	28.1

(continued)

REFERENCE	ECD µm	THICKNESS µm	ASPECT RATIO	RELATIVE SPEED AT 330 nm	RELATIVE SPEED AT 430 nm
13	2.63	0.085	30.94	92.9	19.4

[0063] Figure 3 shows the photographic sensitivity of these films when they are exposed to visible radiation (430 nm) and when they are exposed to ultraviolet radiation (330 nm).

[0064] This figure shows the considerable gain in photographic sensitivity obtained with the films used in the assemblage of the present invention exposed to ultraviolet radiation.

EXAMPLE 4

[0065] The following examples describe double-face radiographic films used in the assemblage the invention with the following structure:

Top layer of gelatin
Intermediate layer
Emulsion
Support
Emulsion
Intermediate layer
Top layer

[0066] The top layer and intermediate layer consist of gelatin (each 3.54 mg/dm^2). The emulsion layer consists of gelatin (32 mg/dm^2) and silver halides (21 mg/dm^2). The film is tanned by means of the compound bis (vinylsulphonylmethyl) ether, the content by weight of tanning agent being equal to 2.35% of the total dry gelatin contained in the film. The emulsions in the films are chemically and spectrally sensitised optimally in accordance with the methods described above.

[0067] In this example, a film was exposed comprising a tabular silver bromide grain emulsion according to the invention (ECD = 2.95 µm, e = 0.16 µm, R = 18.4) and an X-OMAT-K® commercial film (three-dimensional grains) through an X-ray intensifying screen emitting in the ultraviolet range. The exposures were made on a medical radiography table with a SIEMENS POLYDOROS 100® generator equipped with an OPTILIX 150/40/102G® tube with a tungsten rotating anode. The filtration is 2.5 mm of aluminium. The generator operates at 70 kV and between 30 and 40 mA.

[0068] The exposed films are then processed with a conventional development process (X-OMAT-RP® process) and a fast processing method (RA/30®) in accordance with the conditions set out in Table 4 below:

FILM	DEVELOPER	DEVELOPMENT TIME	SPEED
X-OMAT-K®	X-OMAT-RP®	90 sec	100
INVENTION	X-OMAT-RP®	90 sec	103
X-OMAT-K®	RA/30®	45 sec	- *
INVENTION	RA/30®	45 sec	100
X-OMAT-K®	RA/30®	60 sec	- *
INVENTION	RA/30®	60 sec	112

* Results not usable.

[0069] The results show that the films used in the assemblage of the invention may advantageously be processed either using a conventional processing method or a fast processing method, which is not possible with a conventional radiographic film.

EXAMPLE 5

[0070] In this example the films used in the assemblage of the invention (with the double-face format described above) have been compared with the X-OMAT-S® and X-OMAT-K® commercial films described below. In this case the exposures were made on an inverse square bench using a SIEFERT ISOVOLT generator equipped with a 160C tube (tungsten anode) at 70 kV. The quality of the beam was that described in ISO 9236.

REFERENCE	TYPE OF GRAIN	E.C.D. µm	THICKNESS µm
X-OMAT-S®	3D	-	-
X-OMAT-K®	3D	-	-
INVENTION 1	Tabular	2.95	0.16
INVENTION 2	Tabular	2.98	0.13

[0071] The films are exposed between two X-ray intensifying screens, either the X-OMAT-Regular® screen (emission peak 380 nm) consisting of barium sulphate with europium added, or a screen which emits in the ultraviolet wavelengths consisting of germanium oxide and hafnium oxide, the emission of this screen being centred on 340 nm.
[0072] After exposure, the films are developed using an RP X-OMAT MX810® process, at 35°C.
[0073] The sensitometric results obtained with the X-OMATIC Regular® screen are set out in the following table:

REFERENCE	FOGGING	RELATIVE SPEED	CONTRAST
X-OMAT-S®	0.23	100	3.05
X-OMAT-K®	0.22	86	2.61
INVENTION 1	0.23	87	.01
INVENTION 2	0.20	88	3.27

[0074] The sensitometric results obtained with the X-ray intensifying screen emitting in the ultraviolet range are set out in the following table:

REFERENCE	FOGGING	RELATIVE SPEED	CONTRAST
X-OMAT-S®	0.23	100	3.07
X-OMAT-K®	0.22	85	2.60
INVENTION 1	0.23	99	2.76
INVENTION 2	0.20	100	2.99

[0075] These results show the sensitometric performance of the radiographic assemblages of the present invention.

Claims

1. Radiographic assemblage comprising at least one X-ray intensifying screen and a radiographic film consisting of a support with a layer of silver halide emulsion on at least one of its faces, characterised in that at least 50% of the silver halide emulsion consists of tabular grains with a thickness of less than 0.5 µm and an aspect ratio between 5 and 25, and the X-ray intensifying screen comprises a support covered with a fluorescent layer comprising, dispersed in a binder, a luminophore with a maximum emission as close as possible to the intrinsic sensitivity peak of the silver halide grains forming the radiographic film.

2. Radiographic assemblage according to claim 1, in which the thickness of the tabular grains is less than 0.2 µm and the aspect ratio is between 10 and 20.

3. Radiographic assemblage according to Claim 2, in which at least 90% of the silver halide grains are tabular.

4. Radiographic assemblage according to Claim 1, in which the X-ray intensifying screen consists of one or more luminophores in the form of particles in a mixture in a binder transparent to ultraviolet and X-rays.

5. Radiographic assemblage according to claim 4, in which the luminophore or luminophores have a maximum emission between 310 and 350 nm.

6. Radiographic assemblage according to any one of the preceding claims, in which the X-ray intensifying screen consists of particles of germanium oxide and hafnium oxide.

7. Use of a radiographic assemblage according to any one of the preceding claims in a fast processing method.

Patentansprüche

1. Radiografische Anordnung, die mindestens einen Röntgen-Verstärkerschirm und einen Röntgenfilm aufweist, welcher aus einem Träger mit einer Silberhalogenidemulsionsschicht auf mindestens einer seiner Oberflächen aufweist, **dadurch gekennzeichnet**, dass mindestens 50 % der Silberhalogenidemulsion aus tafelförmigen Körnern mit einer Dicke von unter 0,5 µm und einem Abmessungsverhältnis zwischen 5 und 25 besteht, und dass der Röntgenverstärkerschirm einen Träger aufweist, der mit einer fluoreszierenden Schicht bedeckt ist, welche einen in einem Bindemittel dispergierten Luminophor mit maximaler Emission möglichst nahe am Eigenempfindlichkeitsmaximum der den Röntgenfilm bildenden Silberhalogenidkörner aufweist.

2. Radiografische Anordnung nach Anspruch 1, dadurch gekennzeichnet, dass die Dicke der tafelförmigen Körner unter 0,2 µm und das Abmessungsverhältnis zwischen 10 und 20 liegt.

3. Radiografische Anordnung nach Anspruch 2, dadurch gekennzeichnet, dass mindestens 90 % der Silberhalogenidkörner tafelförmig sind.

4. Radiografische Anordnung nach Anspruch 1, dadurch gekennzeichnet, dass der Röntgenverstärkerschirm aus einem oder mehreren Luminophoren in Partikelform besteht, welche sich in einem in eine Mixtur eingebrachten Bindemittel befinden, das für ultraviolette Strahlen und Röntgenstrahlen durchlässig ist.

5. Radiografische Anordnung nach Anspruch 4, dadurch gekennzeichnet, dass der Luminophor oder die Luminophore eine Maximalemission zwischen 310 und 350 nm aufweisen.

6. Radiografische Anordnung nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass der Röntgenverstärkerschirm aus Germaniumoxid- und Hafniumoxid-Partikeln besteht.

7. Verwendung einer radiografischen Anordnung nach einem der vorhergehenden Ansprüche in einem Schnellentwicklungsverfahren.

Revendications

1. Assemblage radiographique comprenant au moins un écran renforçateur de rayons-X et un film radiographique constitué d'un support avec sur au moins une des faces une couche d'émulsion aux halogénures d'argent, carac-

térisé en ce que au moins 50 % de l'émulsion aux halogénures d'argent consiste en des grains tabulaires avec une épaisseur inférieure à 0,5 µm et un indice de forme entre 5 et 25 et l'écran renforçateur de rayons-X comprend un support recouvert d'une couche fluorescente comprenant, dispersé dans un liant, un luminophore ayant une émission maximale aussi proche que possible du pic de sensibilité intrinsèque des grains d'halogénure d'argent formant le film radiographique.

2. Assemblage radiographique selon la revendication 1 dans lequel l'épaisseur des grains tabulaires est inférieure à 0,2 µm et l'indice de forme est compris entre 10 et 20.

3. Assemblage radiographique selon la revendication 2 dans lequel au moins 90 % des grains d'halogénures d'argent sont tabulaires.

4. Assemblage radiographique selon la revendication 1 dans lequel l'écran renforçateur de rayons-X est constitué d'un ou de plusieurs luminophores sous forme de particules en mélange dans un liant transparent aux ultraviolets et aux rayons-X.

5. Système radiographique selon la revendication 4 dans lequel le ou les luminophores ont une émission maximale comprise entre 310 et 350 nm.

6. Système selon l'une quelconque des revendications précédentes dans lequel l'écran renforçateur de rayons-X est constitué de particules d'oxyde de germanium et d'hafnium.

7. Utilisation d'un système radiographique selon l'une quelconque des revendications précédentes dans un procédé de traitement rapide.

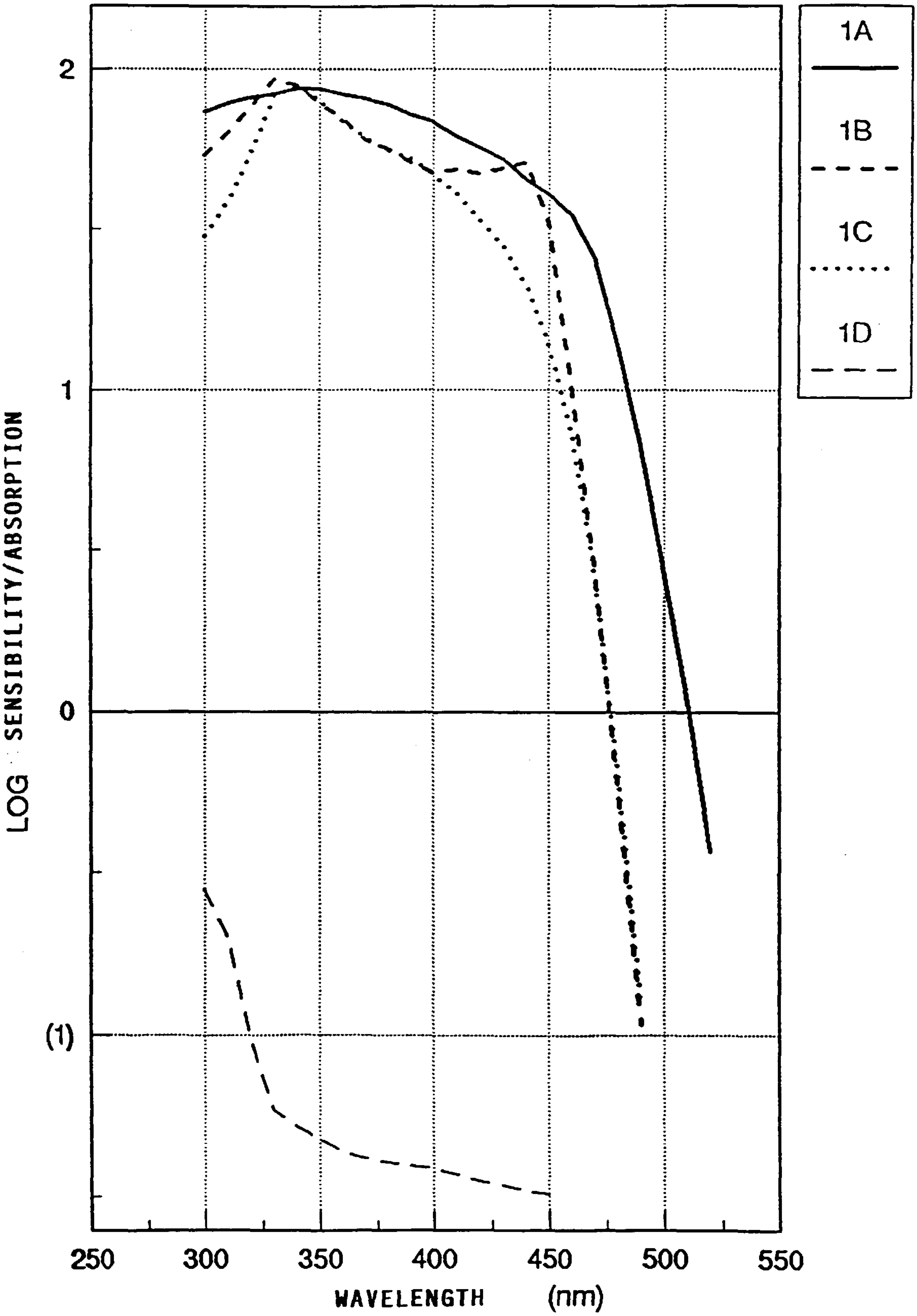
2
1
0
(1)
LOG SENSIBILITY/ABSORPTION
250
300
350
400
450
500
550
WAVELENGTH (nm)
1A
1B
1C
1D

FIGURE 1

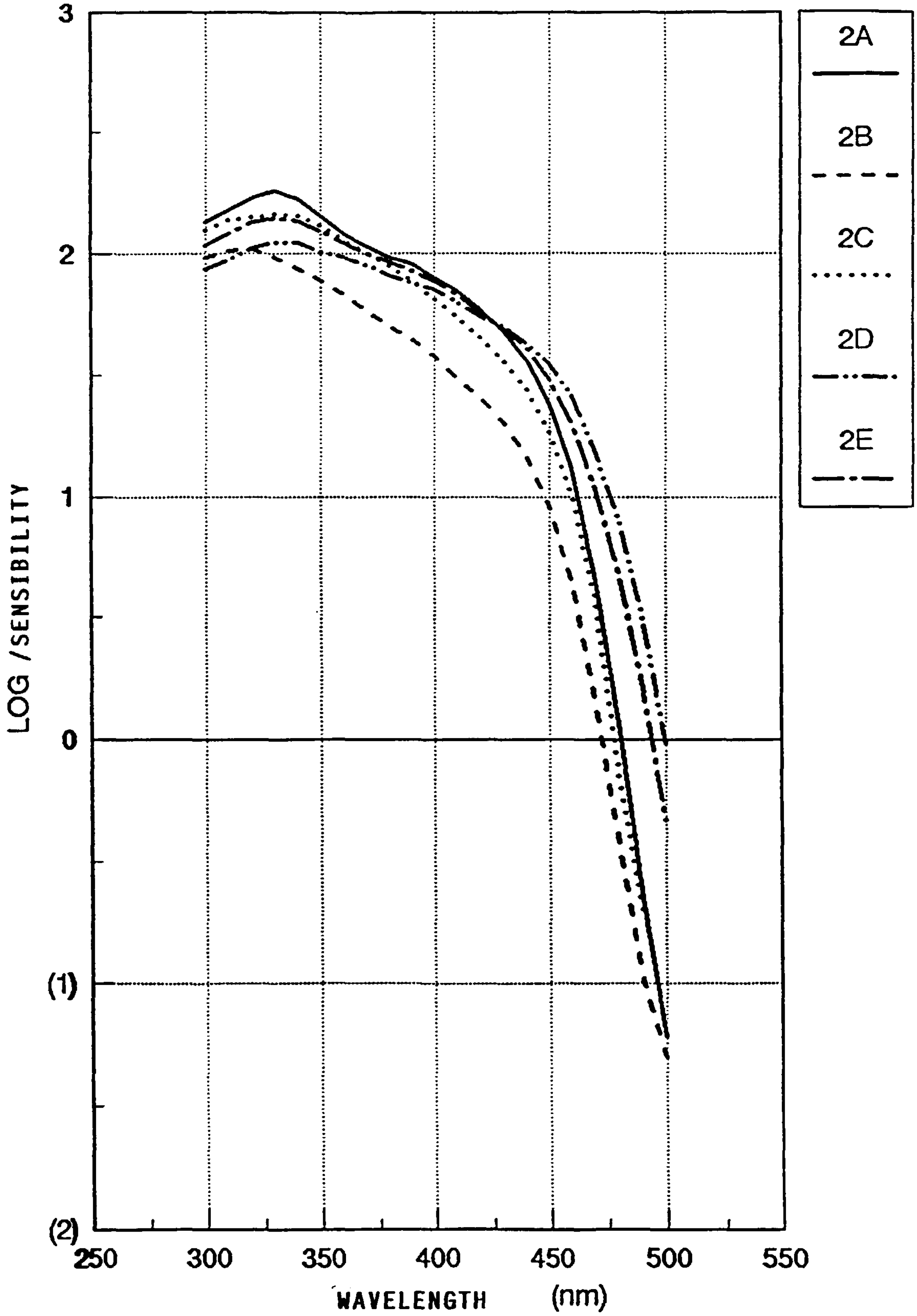
3
2
1
0
(1)
(2)
LOG /SENSIBILITY
250
300
350
400
450
500
550
WAVELENGTH (nm)
2A
2B
2C
2D
2E

FIGURE 2

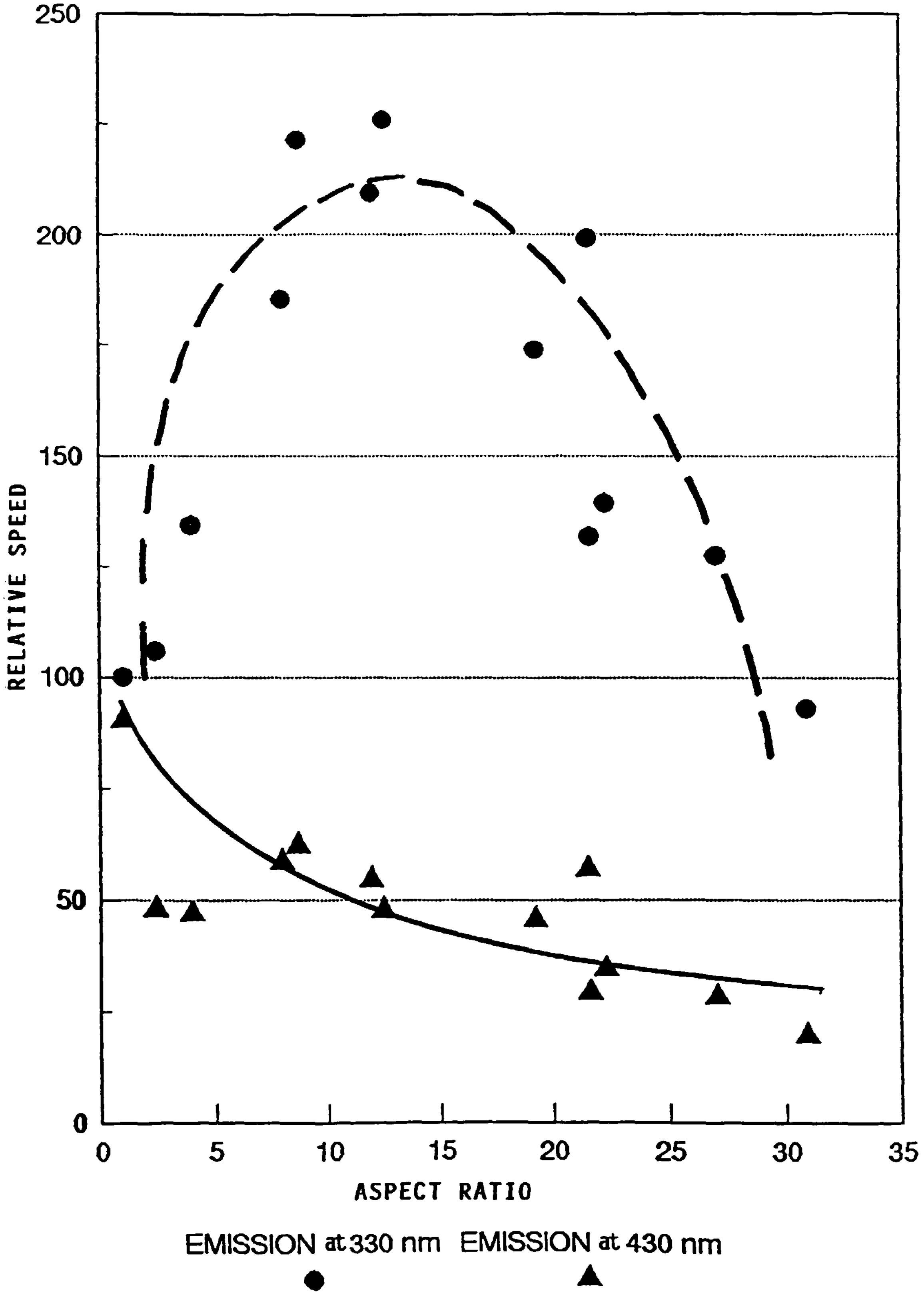
250
200
150
100
50
0
RELATIVE SPEED
0
5
10
15
20
25
30
35
ASPECT RATIO
EMISSION at 330 nm
EMISSION at 430 nm

FIGURE 3